
Section 2

Technology Effectiveness Analysis

This section addresses the effectiveness of Soil Rescue as observed during the demonstration of the technology at the selected sites at the CRPAC. Section 2.1 describes the predemonstration activities that lead to the selection of the two locations for the demonstration; Section 2.2 presents the activities conducted during the demonstration, including the establishment of experimental units at each demonstration site, and the collection of untreated and treated soil samples; Section 2.3 describes the laboratory analytical and statistical methods used to evaluate demonstration objectives; Section 2.4 presents results of the demonstration; and Section 2.5 provides a summary of results obtained from the analysis of quality control samples that were collected during the demonstration.

2.1 PREDEMONSTRATION ACTIVITIES

Predemonstration activities included preliminary sampling at four candidate locations, followed by selection of two demonstration sites. In March 1998, site personnel collected soil samples from four locations that had been identified by OEPA as potential demonstration sites. Three of the locations were at pottery factories, and the other location was at a former trailer park that had been constructed on property contaminated with pottery wastes. At all four locations, field measurements of total lead concentrations were made with an x-ray fluorescence (XRF) analyzer, and additional samples were collected for laboratory analysis of total lead, leachable lead (by the TCLP and SPLP), and soil lead bioaccessibility (by the SIVM). Table 2-1 presents the highest concentrations of lead measured at each of the four locations. The highest concentrations of lead measured in the field by XRF analyzers are higher than those measured in the laboratory because samples for laboratory measurements were not collected at exact locations where the highest field concentrations of lead were detected. As Table 2-1 indicates, the two locations selected for the SITE demonstration were the inactive pottery factory in Roseville, Ohio, and the trailer park, also in Roseville. The principal

reasons for the selection of the inactive pottery factory in Roseville were that it appeared to have higher concentrations of lead than any of the other locations and it was more readily accessible than the other pottery factories. The trailer park was selected for the SITE demonstration primarily because use of that site would allow evaluation of the Soil Rescue technology at sites at which concentrations of lead in soil were lower than those at the pottery factories. At the time the selection was made, there was some concern that the concentrations of lead at the trailer park might be too low because they did not exceed 400 mg/kg, the residential preliminary remediation goal (PRG) for lead established by EPA (EPA 2000). However, previous field sampling conducted by OEPA with XRF analyzers had indicated that total concentrations of lead in the soil at the trailer park were well above 400 mg/kg.

2.2 DEMONSTRATION ACTIVITIES

Section 2.2.1 discusses demonstration activities that were conducted before treatment. Sections 2.2.2 and 2.2.3, respectively, provide detailed descriptions of the demonstration activities that were conducted during and after the demonstration.

2.2.1 Activities Before Treatment

SITE personnel identified a total of 10 experimental units at the trailer park, and only one experimental unit at the inactive pottery factory. All the experimental units were identified through application of the provisions of a judgmental plan based on knowledge of the site and total lead measurements taken with a field XRF.

SITE Program personnel removed the vegetation (sod) from the experimental units. To facilitate the homogenization of the soil and the collection of samples, the soil in the ten experimental units at the trailer park was mixed with a garden tiller to a depth of approximately 6 inches. The soil

Table 2-1. Summary of Maximum Concentrations of Lead Observed During Predemonstration Sampling Activities					
	Maximum Lead Concentrations ¹				
Site Name and Location	Total Field (mg/kg)	Total Laboratory (mg/kg)	Leachable via TCLP (mg/L)	Leachable via SPLP (mg/L)	Bioaccessible via SIVM (%)
Trailer Park, Roseville, Ohio ²	300	134	32.0	<0.50	47
Inactive Pottery Factory, Roseville, Ohio ²	23,100	8,170	48.6	<0.50	31
Active Pottery Factory, Roseville, Ohio	14,500	1,080	57.9	<0.50	42
Inactive Pottery Factory, Crooksville, Ohio	2,654	793	77.1	<0.50	76
¹ The results reported represent the maximum concentrations detected, rather than a single sample from any one location. Total lead measurements in the field were made with XRF analyzers; total lead measurements in the laboratory were made by nitric acid digestion (SW-846 3050B). TCLP = toxicity characteristic leaching procedure; SPLP = synthetic precipitation leaching procedure; SIVM= simplified in-vitro method). ² The trailer park and the inactive pottery factory, both located in Roseville, Ohio, were selected for the SITE demonstration.					

in the one experimental unit at the inactive pottery factory was homogenized by mixing soil with a backhoe to a depth of 6 inches. The 10 experimental units in the trailer park were assigned letters (C,G,K,L,M,N,O,Q,R,T), as was the experimental unit adjacent to the inactive pottery factory (U). Each of the 10 units in the trailer park measured 5 feet wide by 5 feet long, and the single unit at the inactive pottery factory unit measured 3 feet wide by 6 feet long. The depth of the demonstration in all units was limited to the upper 6 inches of soil. Figure 2-1 shows the locations of the experimental units at the trailer park, and Figure 2-2 shows the location of the experimental unit at the inactive pottery factory.

To establish the conditions present before the application of Soil Rescue, soil samples were collected from each experimental unit. However, the samples were collected differently at the two locations. At the trailer park, composite samples were collected from each of the 10 experimental units; at the inactive pottery factory, five grab samples were collected from the single experimental unit. Specific sampling procedures are described below for the trailer park and the inactive pottery factory.

The composite soil samples for each experimental unit at the trailer park were prepared by collecting an aliquot of soil from each corner and from the middle of the experimental unit, as Figure 2-1 shows. Each aliquot was

placed in a stainless-steel bowl (approximate volume: 64 ounces) with a stainless steel spoon or trowel. The technology was not to be evaluated for its ability to treat pottery chips; therefore, the soil samples were screened through a brass 3/8-inch sieve into a plastic 5-gallon bucket to remove pottery chips from the samples. Particles larger than 3/8 inch were returned to the stainless steel bowl, and the percentage of the particles, on the basis of volume, that did not pass through the sieve was estimated and recorded in the logbook. The composite sample was hand-mixed in the bucket with a stainless-steel spoon for one minute before the sample containers were filled. After mixing, fractions for the various analyses were prepared by filling the sample containers with the composited soil. Field duplicate samples were collected from two of the experimental units at the trailer park.

The five grab soil samples collected from the single experimental unit at the inactive pottery factory were collected before treatment from each corner and the from middle of the experimental unit, as shown in the inset diagram on Figure 2-2. Each grab soil sample was placed in a separate stainless-steel bowl (approximate volume: 64 ounces) with a stainless-steel spoon or trowel. The grab soil sample was sieved through a brass 3/8-inch sieve into a plastic 5-gallon bucket. Particles larger than 3/8 inch were returned to the stainless steel bowl, and the percentage of the particles, on the basis of volume, that did not pass

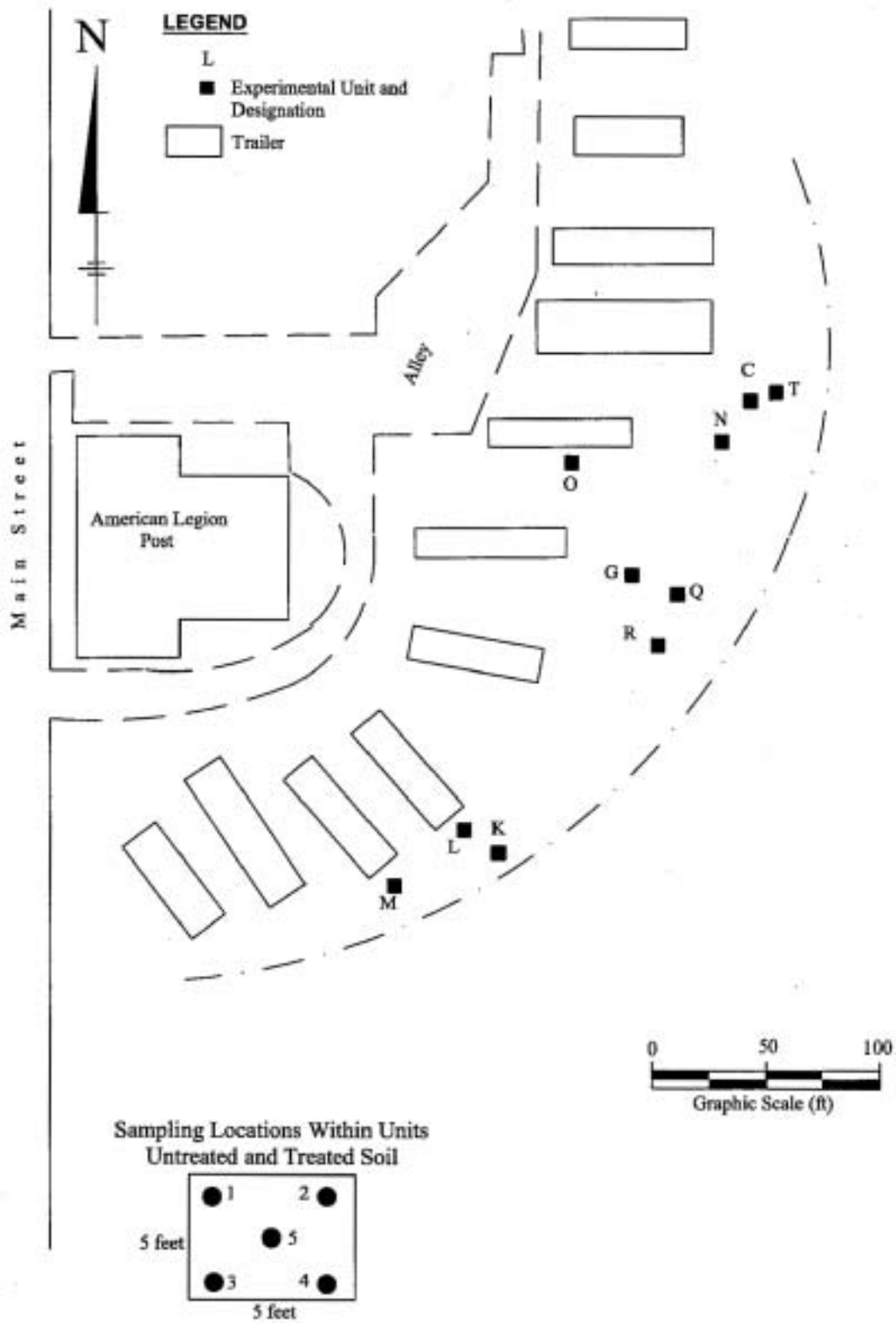
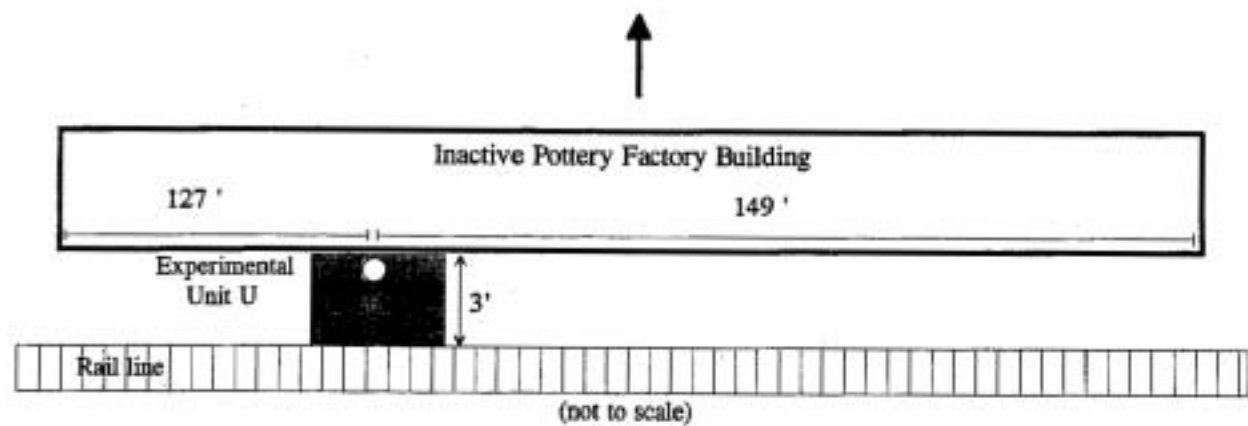
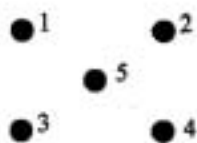


Figure 2-1. Trailer park sampling locations and patterns.

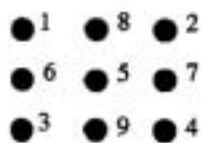


Sampling Locations for Untreated Soil



Experimental Unit U

Sampling Locations for Treated Soil



Experimental Unit U

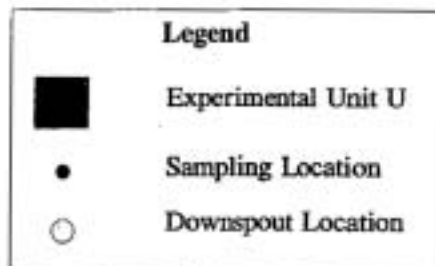


Figure 2-2. Inactive pottery factory sampling locations and patterns.

through the sieve was estimated and recorded in the logbook. Each grab sample was hand-mixed in the bucket with a stainless-steel spoon for one minute before the sample containers were filled. The grab samples from various locations were not composited. One field duplicate sample was collected from one of the grab soil samples in one of the sampling buckets.

2.2.2 Treatment Activities

After completing the activities described in Section 2.2.1, Star Organics, using a pressurized wand, applied Soil Rescue to the soil in each experimental unit to a depth of two feet.

2.2.3 Activities After Treatment

SITE personnel evaluated the effectiveness of the treatment by collecting and analyzing soil samples after the technology was applied and comparing the data from those samples with the data on the untreated soil. Soil samples were collected from the experimental units treated with Soil Rescue after a minimum of 72 hours after treatment. Sampling of treated soils at the trailer park consisted of collecting and compositing five soil aliquots from each experimental unit in the same manner in which the samples of untreated soil were collected. At the inactive pottery factory, grab samples of treated soils were collected from the single experimental unit in the same manner in which the samples of untreated soil were collected, except that nine grab samples were collected instead of five (see Figure 2-2) to obtain a more precise estimate of the treated sample mean.

2.3 LABORATORY ANALYTICAL AND STATISTICAL METHODS

The SITE program samples collected during the demonstration were analyzed by methods described in the QAPP approved by EPA (Tetra Tech EM Inc. [Tetra Tech] 1998). Statistical analyses were performed on selected analytical data to demonstrate whether the criteria set forth in the primary and secondary objectives were met. The following section presents a brief description of the analytical procedures and statistical methods used to evaluate the samples that were collected during the demonstration.

2.3.1 Laboratory Analytical Methods

Several analytical methods were used to evaluate the project objectives on the basis of the specific analyses of interest and the minimum detectable concentrations needed

to achieve the project objectives. Whenever possible, methods approved by EPA were selected to analyze the soil samples collected during the demonstration. The following references were used in performing the standard analytical procedures approved by EPA:

- EPA. 1996. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Laboratory Manual, Volume 1A through 1C and Field Manual, Volume 2, SW-846, Third Edition, Update III. EPA Document Control No 955-001-00000-1. Office of Solid Waste Washington, DC, December. (For convenience, analytical methods from this reference are referred to as SW-846, followed by their respective analytical method number.)
- EPA. 1983. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 and subsequent EPA-600/4-technical additions. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio. (For convenience, analytical methods from this reference are referred to as MCAWW followed by their respective analytical method number.)

When standard methods were not available, or when the standard methods did not meet the project objectives, other published methods were used to analyze the soil samples. The nonstandard methods were evaluated and approved for use by EPA NRMRL before the soil samples were analyzed. Table 2-2 lists the parameters, matrices, method references, and method titles for the analytical laboratory procedures used to evaluate the SITE demonstration samples. Brief descriptions of the extraction procedures, lead analytical procedures, and nonstandard analytical procedures used in the demonstration are provided below.

Standard Extraction Procedures

Three standard extraction procedures approved by EPA were used to analyze soil samples to determine the concentrations of lead that will leach under various conditions – the TCLP, the MEP, and the SPLP. The TCLP is used to determine the mobility of contaminants in solids and multiphase waste; it simulates the initial leaching that a waste would undergo in a sanitary landfill. The MEP was designed to simulate both the initial and the subsequent leaching that a waste would undergo in an improperly designed sanitary landfill, where it would be subjected to prolonged exposure to acid precipitation. The SPLP is designed to simulate the initial leaching that a waste would undergo if it were disposed of in a monofill, where it would be subjected to exposure to acid precipitation (EPA 1996). The multiphase steps in performing the extraction procedures are described below.

Table 2-2. Analytical Laboratory Methods			
Parameter	Matrix	Method Reference	Title of Method
TCLP Lead	Soil	SW-846 1311	Toxicity Characteristic Leaching Procedure
Soil Lead Bioaccessibility	Soil	SIVM (SBRC 1998)	In Vitro Method for Determination of Lead and Arsenic Bioaccessibility
MEP Lead	Soil	SW-846 1320	Multiple Extraction procedure
Lead Speciation by Scanning Electron Microscopy	Soil	Standard Operating Procedure for Metal Speciation (University of Colorado 1998)	Standard Operating Procedure for Metal Speciation (Draft)
Lead Speciation by Sequential Soil Serial Extractions	Soil	Sequential Extraction Procedure for the Speciation of Particulate Trace Metals (Tessier 1979)	Sequential Extraction Procedure for the Speciation of Particulate Trace Metals
Eh	Soil	SW-846 9045C	Soil and Waste pH
pH	Soil	SW-846 9045C	Soil and Waste pH
CEC	Soil	Soil Sampling and Methods of Analysis (Canadian Society of Soil Science 1993)	Exchangeable Cations and Effective CEC by the BaCl ₂ Method
Acid Neutralization Capacity	Soil	Environment Canada Method No. 7	Acid Neutralization Capacity
Total Lead using Nitric Acid Digestion	Plants, Water, Filters	SW-846 3050B, followed by SW-846 6010B	Acid Digestion of Sediments, Sludges, and Soils, Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)
Oil and Grease	Soil	EPA Method 1664	Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons)
Total Lead Hydrofluoric Acid Digestion	Soil	SW-846 3052, followed by SW-846 6010B	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices, Inductively Coupled Plasma-Atomic Emission Spectrometry
SPLP Lead	Soil	SW-846 1312	Synthetic Precipitation Leaching Procedure
Phosphates	Soil	SW-846 9056	Determination of Inorganic Anions by Ion Chromatography
Humic and Fulvic Acid	Soil	Soil Sampling and Methods of Analysis (Canadian Society of Soil Science, 1993)	Soil Humus Fractions
Soil Classification	Soil	ASTM D2487-93	Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)
VOCs	Soil	SW-846 8260B	Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometry
SVOCs	Soil	SW-846 8270C	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry: Capillary Column Technique

The basic steps in performing the extraction procedures are:

- Determine the appropriate solution by reviewing preliminary analyses of the soil's solid content and pH of the soil
- Prepare the appropriate extraction fluid (consisting of one or more concentrated acids, depending on the procedure), diluted with distilled deionized water
- Place a specified quantity of the soil sample in an extraction vessel with a predetermined quantity of extraction fluid
- Rotate the vessel at the specified rotations per minute (rpm) for the appropriate amount of time (18 to 24 hours)
- Maintain the temperature as described in the methods
- Separate the material by filtering the content of the vessel through a glass fiber filter
- Analyze the resulting liquid for lead concentrations of lead by the procedures set forth in SW-846 methods 3050B and 6010B

Extraction Procedure for Bioaccessible Lead

The extraction procedure for soil lead bioaccessibility is presented in the SIVM. The steps in the procedure are:

- Air dry the soil sample, grind it with a mortar and pestle, and sieve it with a less than 250 microns (μm) sieve
- Analyze the sample for total lead using a XRF analyzer
- Add the sample to an aqueous extraction fluid consisting of deionized water, glycine as a buffer, and concentrated hydrochloric acid

- Maintain the sample and extraction fluid at a pH of $1.50, \pm 0.05$, and tumble both in a water bath at 37°C for one hour, using a modified TCLP apparatus
- Collect 15 milliliters (mL) of extract from the extraction vessel into a 20-cubic-centimeter syringe and filter through a 0.45-micrometer (μm) cellulose acetate disk filter into a 15-mL polypropylene centrifuge tube
- Analyze the filtered extract for lead using ICP-AES according to SW-846 Method 6010B

Table 2-3 summarizes the acids used in extraction fluids and other operational parameters of the extraction procedures.

Lead Speciation by Scanning Electron Microscopy

The percent frequency of various lead species (hereafter referred to as lead phases) in soil samples before and after treatment was determined by application of the metal speciation procedure developed by Dr. John Drexler (University of Colorado 1998). The procedure uses an electron microprobe (EMP) technique to determine the frequency of occurrence of metal-bearing phases in soil samples.

The EMP used for this analysis is equipped with four wavelength dispersive spectrometers (WDS), an energy dispersive spectrometer (EDS), a backscatter electron imaging (BEI) detector for taking photomicrographs, and a data processing system. Two of the spectrometers were equipped with synthetic "pseudocrystals" that have been developed recently for WDS applications. The pseudocrystals are known as layered dispersive elements (LDE). The materials are composed of alternating layers of boron and molybdenum of varying thicknesses and are designed to optimize the separation of individual wavelengths in the x-ray characteristic radiation spectrum. The first of

Method	Extraction Fluid	pH of Fluid	Temperature	Time of Extraction
TCLP	Acetic acid	4.93 ± 0.05	$23^\circ\text{C} \pm 2^\circ\text{C}$	18 ± 2 hours
MEP (first extract)	Acetic acid	5.0 ± 0.2	$20^\circ\text{C} - 40^\circ\text{C}$	24 hours
MEP (second through ninth extracts)	Sulfuric and nitric acids	3.0 ± 0.2	$20^\circ\text{C} - 40^\circ\text{C}$	24 hours
SPLP	Sulfuric and nitric acids	4.20 ± 0.05	$23^\circ\text{C} \pm 2^\circ\text{C}$	18 ± 2 hours
SIVM	Hydrochloric acid	1.50 ± 0.05	37°C	1 hour

the materials to be produced for WDS applications (LDE-1) was used in one of the spectrometers for the determination of oxygen. Another spectrometer was equipped with a LDE designed to detect carbon (LDE-C).

Lead speciation was determined by using the EMP to perform point counts on the samples. Point counting is a method of determining the volume fractions of constituent phases in a sample from the relative areas, as measured on a planar surface. The EMP analyzes a sample on a point-by-point basis to determine how much of a given phase is present in a sample. The point counts were performed by crossing each sample from left to right and from top to bottom with the electron beam. The amount of vertical movement for crossing depends on the magnification used and the size of the cathode-ray tube. In all cases, the movement was kept to a minimum so that no portion of the sample was missed. Two magnification settings were used for each sample, one ranging from 40 to 100 X and the other ranging from 300 to 600 X. The second magnification allowed the identification of the smallest identifiable phases (1 to 2 μm). The precision of the EMP lead speciation data was determined from duplicate analysis performed every 20 samples.

Lead Speciation by Sequential Extractions

The lead phases in the soil samples from both sites were identified by application of Tessier's sequential extraction procedure (Tessier 1979). The soil samples were analyzed by the Laboratory for Environmental and Geological Studies at the University of Colorado, Boulder.

The soil samples were air-dried, ground with a mortar and pestle, and sieved to less than 250 μm . The procedure uses sequential chemical extractions with different reagents to determine the concentration of lead that partitions into each of several discrete metal phases. The phases include exchangeable lead, lead bound to carbonates, lead bound to iron oxide, lead bound to manganese oxide, lead bound to organic matter, and residual lead. Approximately one gram of the sample aliquot (dried weight) was used for the initial extraction. The reagent used to extract the exchangeable lead phase was magnesium chloride (MgCl_2) at a pH of 7.0. For the second extraction, a solution of sodium acetate and acetic acid at a pH of 5.0 was used to extract the lead bound to carbonates. For the third extraction, a hydroxyl amine hydrochloride in 25 percent acetic acid (pH \sim 2) solution was used to extract the lead bound to iron and manganese oxides. For the fourth extraction, hot hydrogen peroxide in a nitric acid solution and subsequently ammonium acetate were used to extract

the lead bound to organic matter. For the final extraction, a solution of hydrofluoric and perchloric acid solution was used to extract the lead bound to primary and secondary minerals (the residual phase).

Oxidation-Reduction Potential

The soil samples were prepared for determining Eh using the sample preparation procedures set forth in SW-846 Method 9045C. The method consisted of preparation of a soil suspension by adding 20 mL of reagent water to 20 grams of soil. The mixture was covered and stirred for five minutes. The soil suspension was allowed to stand for one hour to allow most of the suspended clay to settle out of the suspension. The Eh then was measured according to American Society for Testing and Materials (ASTM) Test Method D1498-93, "Standard Practice for Oxidation-Reduction Potential of Water." A meter capable of reading millivolts (mV) with a reference electrode and an oxidation-reduction electrode was used to take the measurements. The meter first was allowed to warm up for two to three hours before measurements were taken. After the meter was checked for sensitivity and the electrodes were washed with deionized water, the electrodes were placed into the sample. While the sample was agitated with a magnetic stir bar, successive portions of the sample were measured until two successive portions differed by no more than 10 mV.

pH

The pH was evaluated by application of the procedures set forth in SW-846 Method 9045C. The method consisted of the preparation of a soil suspension by adding 20 mL of reagent water to 20 grams of soil. The mixture was covered and stirred for five minutes. The soil suspension was allowed to stand for one hour to allow most of the suspended clay to settle out of the suspension. A pH meter was allowed to warm up for two to three hours before measurements were taken. After the meter was checked for sensitivity and the electrodes were washed with deionized water, the electrodes were placed in the clear supernatant portion of the sample. If the temperature of the sample differed by more than 2EC from that of the buffer solution, the pH values measured were corrected for the temperature difference.

Cation Exchange Capacity

One sample from the untreated and treated soil samples from each site was selected for evaluation of CEC, which was determined by the barium chloride (BaCl_2) method. The BaCl_2 method provides a rapid means of determining

the exchangeable cations and the “effective” CEC of a wide range of soil types. By that method, CEC is calculated as the sum of exchangeable cations (Ca, Mg, K, Na, Al, Fe, and Mn). The procedure consisted of the following steps:

- The soil sample was air-dried, ground using a mortar and pestle, and sieved to less than 250 μm
- Approximately 0.5 gram of soil was placed into a 50-mL centrifuge tube with 30.0 mL of 0.1 molar BaCl_2 , and the mixture was shaken slowly on an end-over-end shaker at 15 rpm for 2 hours
- The mixture was centrifuged for 15 minutes, and the supernatant portion was filtered through a Whatman No. 41 filter paper
- The cations were analyzed with an atomic absorption spectrophotometer

Acid Neutralization Capacity

The acid neutralization capacity of the soil was determined by application of Environment Canada Method No. 7. The soil sample was air-dried, ground using a mortar and pestle, and sieved to less than 250 μm . The amount of neutralizing bases, including carbonates, was then determined by treating each sample with a known excess of standardized hydrochloric acid. The sample and acid were heated to allow completion of the reaction between the acid reagent and the neutralizers in the soil sample. The calcium carbonate equivalent of the sample was obtained by determining the amount of unconsumed acid by titration with standardized sodium hydroxide.

Lead Analytical Procedures

Two procedures were used to determine the lead concentrations in the soil. One analytical procedure used a nitric acid solution to measure all but the most stable forms of lead in the sample, and the other procedure used hydrofluoric acid to measure all of the lead in the sample. The nitric acid digestion procedure involved digesting approximately one gram of soil with a solution of nitric acid, hydrogen peroxide, and hydrochloric acid. The mixture was heated to 95°C, \pm 5°C, for approximately two hours. The digestate was filtered through Whatman No. 41 filter paper into a flask and analyzed for lead ICP-AES, as described in SW-846 Method 6010B.

The hydrofluoric acid digestion procedure involved heating approximately one gram of soil in a solution containing nitric and hydrofluoric acids to 180°C, \pm 5°C, for approximately 9.5 minutes. The digestate was filtered through Whatman No. 41 filter paper into a flask, and the

filtrate was analyzed for lead by ICP-AES, as described in SW-846 Method 6010B.

Soil Classification

Soil classification consisted of determining the particle size distribution, liquid limit, and plasticity index of the soil samples. That information was used to classify the soil according to basic soil group, assigning a group symbol and name. The particle size distribution was determined by sieving the dried soil samples through a series of sieves and determining the percentage by weight that was retained on the sieves. The liquid limit is the water content (measured as percent moisture) at which a trapezoidal groove cut in moist soil (in a special cup) closes after being tapped 25 times on a hard rubber plate. The plastic limit is the water content at which the soil breaks apart when rolled by hand into threads of 1/8-inch diameter. The plasticity index is determined by first determining the liquid and plastic limits and then subtracting the plastic limit from the liquid limit.

Humic and Fulvic Acids

Humic and fulvic acids were extracted from the soil samples and quantified through the use of a sodium hydroxide solution, as described below:

- Air dry 15 g of soil, grind it to less than 250 μm , and place it in a 250-mL plastic centrifuge bottle
- Add 150 mL of 0.5 molar hydrochloric acid, let the mixture sit for one hour, and then centrifuge it for 15 minutes and discard the supernatant portion
- Add 150 mL of deionized water to the centrifuge bottle and mix it to wash the soil of remaining acid; centrifuge again for 15 minutes and discard the supernatant portion
- Add 150 mL of 0.5 molar sodium hydroxide to the centrifuge bottle and flush the head space with oxygen-free nitrogen gas
- Place the bottle on an end-over-end shaker for 18 hours
- Centrifuge the mixture for 15 minutes, decant the supernatant portion, and separate that portion into the humic and fulvic fractions by acidifying the extract to a pH of 1.5; the precipitate is the humic acid fraction, and the supernatant portion is the fulvic acid fraction

2.3.2 Statistical Methods

This section provides a brief overview of the statistical methods that were used to evaluate the data from the SITE demonstration. The methods included assessing the

distribution of sample data and calculating specific parametric and distribution-free statistics.

2.3.2.1 Determination of the Distributions of the Sample Data

A preliminary assessment of distribution of data was conducted to determine the approximate statistical distribution of the sample data when parametric hypothesis tests were performed. For the evaluation of the data collected for the primary and secondary objectives, sample data distributions were determined by the following methods: (1) common graphical procedures, including histograms, box-plots, stem-and-leaf plots, and quartile-quartile plots, and (2) formal testing procedures, such as the Shapiro-Wilk test statistic, to determine whether a given data set exhibits a normal distribution.

2.3.2.2 Parametric and Distribution-Free Test Statistics

Various testing procedures were employed to determine whether there were any significant differences between concentrations of lead and concentrations of other analytes of interest in the treated soil and the untreated soil. Table 2-4 summarizes the statistical procedures used in evaluating the analytical results associated with each of the objectives of the SITE demonstration. As the table shows, all the parametric statistical procedures used to evaluate the data from the demonstration involved the Student's t-tests. Paired Student t-tests were conducted on data collected from the trailer park, and unpaired Student t-tests were required on data from the pottery factory because of the unequal sizes of samples of treated and untreated soils from that location (see Figure 2-2). In addition, the formula for the Student's t-test was adjusted for evaluation of P2, because the estimator used for that objective (percent reduction of percent bioavailable lead) required manipulation to avoid the creation of a Cauchy (nonnormal) distribution, which cannot be evaluated by a Student's t-test. Data points obtained from the trailer park for evaluation of P2 (sufficient data from the pottery factory were not available for application of a meaningful Student's t-test for evaluation of P2) were evaluated in a paired Student's t-tests, using the following formula:

$$y_i = x_{ti} - x_{ui} \quad , \quad y_m = \sum_{i=1}^n y_i / n \quad , \quad \text{and} \quad S_y^2 = \sum_{i=1}^n (y_i - y_m)^2 / (n - 1)$$

where x_{ti} and x_{ui} represent the i th observations about treated and untreated soils, n represents the sample size, y_i represents the calculated difference between the i th observations, y_m represents the arithmetic mean of the

calculated differences, and S_y^2 represents the calculated variance.

The calculation results in the following t-test statistic:

$$t = \frac{y_m}{\sqrt{(S_y^2) / n}}$$

which follows a t-distribution with $n-1$ degrees of freedom. The test then can be used to determine whether the observed mean difference varies significantly from 0.

The formula used for testing for a $100(1-r_0)$ percent reduction in the arithmetic mean contaminant levels between normally distributed (paired) data on treated and untreated soils for P2 was:

$$\hat{C}_R = C_T - C_U(1 - r_0) \quad \text{where} \quad C_T = \sum_{i=1}^n x_{ti} / n \quad \text{and} \quad C_U = \sum_{i=1}^n x_{ui} / n$$

where x_{ti} and x_{ui} represent the i th observations about the treated and untreated soils, n represents the sample size, C_T and C_U represent the arithmetic mean of observations about the treated and untreated soils, r_0 represents the proportionality reduction factor (for example, if testing for a 25 percent reduction, $r_0 = 0.25$), and \hat{C}_R represents the computed test statistic. The variance for the estimate was calculated as follows:

$$\text{Var}(\hat{C}_R) = [S_T^2 + (1 - r_0)^2 S_U^2 - 2(1 - r_0) S_{UT}] / n$$

where S_T^2 and S_U^2 represent the calculated sample variance for the treated and untreated soils, S_{UT} represents the calculated sample covariance between the soils, and the term $\text{Var}()$ symbolizes "the variance of." However, the following more convenient calculation was applied to the individual, paired observations:

$$y_i = x_{ti} - (1 - r_0) x_{ui} \quad , \quad y_m = \sum_{i=1}^n y_i / n \quad , \quad \text{and} \quad S_y^2 = \sum_{i=1}^n (y_i - y_m)^2 / (n - 1)$$

where all terms are defined as before, since it can be easily shown that:

$$y_m = \hat{C}_R \quad \text{and} \quad S_y^2 = \text{Var}(\hat{C}_R).$$

That calculation resulted in the following t-test statistic:

$$t = \frac{y_m}{\sqrt{(S_y^2) / n}}$$

which follows a t-distribution with $n-1$ degrees of freedom.

Bootstrap resampling analysis, a distribution-free analysis, was performed when assumptions about the distribution of the sample data were not met. Bootstrap resampling was used to estimate means, confidence intervals, or construct hypothesis tests. Bootstrap resampling techniques also

Table 2-4. Summary of Statistical Procedures Used to Evaluate Each of the Objectives of the Demonstration		
Objective	Test Method/ Test Variable	Statistical Method/Acceptance Criterion for Meeting the Objective
P1: Determine whether leachable lead in soil can be reduced to concentrations that comply with the alternative UTS for contaminated soil that are codified at 40 CFR part 268.49 ¹ .	TCLP/Mean concentration of lead in extract (mg/L)	Student's t-test formula at the 0.05 level of significance/Mean concentration of the treated soil must be less than 7.5 mg/L or 90 percent of the mean concentration in untreated soil, whichever is the higher value.
P2: Determine whether the portion of total lead in soil that is "bioaccessible," as measured by an experimental method, can be reduced by at least 25 percent ² .	SIVM/Mean percentage of total lead extracted by the method	Student's t-test formula at the 0.05 level of significance/Mean percentage of total lead in the extract from the treated soil must be at least 25 percent lower than the mean percentage of total lead in the extract from the untreated soil.
S1: Evaluate the long-term chemical stability of the treated soil.	MEP/Mean lead concentration in each extract (mg/L)	Review of test results/Concentrations of all extracts from the treated soils must be lower than 5 mg/L (a nominal concentration that would be expected to meet or exceed cleanup goals at some sites).
	SEM lead speciation/Percent distribution of lead among various lead phases ³	Review of test results/Percent frequencies of more soluble and less soluble phases of lead in the treated and untreated soils must be lower and higher, respectively.
	Sequential extraction/Mean concentration of lead in each phase (mg/L)	Student's t-test formula at the 0.05 level of significance/Mean concentrations of the more soluble and less soluble phases of lead in the treated and untreated soils must be lower and higher, respectively.
	Eh (mV)	Student's t-test formula at the 0.05 level of significance/Mean Eh of the treated soil must be lower than that of the untreated soil.
	pH	Student's t-test formula at the 0.05 level of significance/Mean pH of the treated soil must be higher than that of the untreated soil and 7.0.
	CEC/Milliequivalents per gram (meq/g)	Review of test results/CEC must be increased, as indicated by a qualitative review of statistical summary data.
	Acid neutralization capacity/meq/g	Review of test results/Neutralization capacity must be increased, as indicated by a qualitative review of statistical summary data.
	Total lead–nitric acid/Mean lead concentration of lead (mg/kg)	Student's t-test formula at the 0.05 level of significance/Mean concentration of lead in the treated soil must be lower than that in the untreated soil.
(continued)		

Table 2-4. Summary of Statistical Procedures Used to Evaluate Each of the Objectives of the Demonstration (continued)		
Objective	Test Method/ Test Variable	Statistical Method/Acceptance Criterion for Meeting the Objective
	Total lead–hydrofluoric acid /Mean concentration of lead (mg/kg)	Student's t-test formula at the 0.05 level of significance/Mean concentration of lead in the treated soil must not be higher or lower than that in the untreated soil.
	SPLP lead/Mean concentration of lead in the extract (mg/L)	Student's t-test formula at the 0.05 level of significance/Mean concentration of lead in the extract of the treated soil must be less than 5 mg/L (a nominal concentration that would be expected to meet or exceed cleanup goals at some sites).
	Total phosphate/Mean concentration of phosphate	Review the results/Mean concentration of total phosphates in the treated soil must not be significantly higher or lower less than that in the untreated soil.
	SPLP phosphate/Mean concentration of phosphate in the extract (mg/L)	Review the results/Mean concentration of phosphate in the extract of the treated soil must be less than or equal to that of the untreated soil.
S2: Demonstrate that the application of Soil Rescue did not increase the public health risk of exposure to lead.	Total lead/Mean concentration of lead in the air (mg/m ³)	Review of test results/Concentrations of airborne lead must not exceed NAAQS limits for lead.
S3: Document baseline geophysical and chemical conditions in the soil before the application of Soil Rescue.	Soil classification, total VOCs, SVOCs, oil and grease, and humic and fulvic acids	Review of test results/Identify results that appear unusual in light of the location and history of the site (no specific acceptance criteria were established for S3).
S4: Document operating and design parameters for Soil Rescue.	Cost analyses	Present cost data/No specific acceptance criteria were established for S4.
Notes: ¹ Objective P1 was evaluated statistically only on analytical results from the inactive pottery factory; only three samples pertinent to that objective were collected from the trailer park. ² Achievement of P2 was evaluated only at the trailer park. ³ SEM lead speciation was conducted only on soils collected from the trailer park.		

were used to check the results produced by various parametric tests. A bootstrap analysis was performed on the soil lead bioaccessibility data on the paired samples. The bootstrap analysis was performed by drawing N samples of size n from the observed individual percent reduction (PR) sample values defined as:

$$PR_i = 100 \left(1 - \frac{x_{ti}}{x_{ui}} \right)$$

where x_{ti} and x_{ui} once again represent the i th observations about treated and untreated soils, n represents the sample size, and N represents the number of times the simulations were performed ($N = 1000$ and $n = 10$ for this study). The bootstrap samples then were used to calculate: (1) the observed mean percent reduction; (2) a 100(1-alpha)% confidence interval for this mean estimate, using the observed bootstrap cumulative distribution function; and (3) the proportion of sample means that exceed a given 100(1- r_0)% threshold (that calculation represents a bootstrap version of a hypothesis test).

2.4 RESULTS OF THE SITE DEMONSTRATION

The following sections present the analytical data relevant to each objective of the demonstration and the results of evaluations of those data, including summaries of statistical calculations. Section 2.4.1 addresses P1, Section 2.4.2 addresses P2, and sections 2.4.3 through 2.4.6 address S1 through S4, respectively.

2.4.1 Evaluation of P1

Determine whether leachable lead in soil can be reduced to concentrations that comply with the alternative UTS for contaminated soil that are codified at 40 CFR part 268.49.

The treatment standards for contaminated soil that are codified at 40 CFR part 268.49 require that the concentrations of lead in the treated soil, as measured by the TCLP, must be less than 7.5 mg/L or at least 90 percent lower than those in the untreated soil, whichever is the

higher concentration. Soil samples were collected from the experimental unit at the inactive pottery factory before and after treatment to assess the Soil Rescue treatment process. Table 2-5 summarizes the TCLP lead data for the inactive pottery factory site.

The results of the statistical analysis of those data, shown in Table 2-6, demonstrate that the mean concentration of TCLP lead in treated soil from the inactive pottery factory was significantly less than 7.5 mg/L; in fact, the results reflect a probability of less than 0.005 (or 1 in 500) that the actual mean concentration of TCLP lead in the treated soils is higher than 7.5 mg/L. Therefore, it was concluded that Soil Rescue achieved the first primary objective (P1) of the SITE demonstration. In addition, Soil Rescue exceeded P1 in that the mean concentration of TCLP lead in the untreated soil was reduced by more than 99 percent.

Data from the trailer park were not used to evaluate P1 on a formal statistical basis; however, concentrations of TCLP lead were measured in untreated and treated soil at 3 of the 10 experimental units at that location. The analytical results for TCLP lead from two of those experimental units indicate similar reductions in concentrations of TCLP lead. No reductions in concentrations of TCLP lead could be identified for samples collected at the third experimental unit, because the concentrations of TCLP lead in both untreated and treated soils from that unit were lower than detection limits. Table 2-7 summarizes the TCLP lead results from the trailer park.

Table 2-5. TCLP Lead Results for the Inactive Pottery Factory Site			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
U	1	453	3.2
U	2	376	3.0
U	3	411	3.6
U	4	364	3.5
U	5	411	3.1
U	6	n/s	4.0
U	7	n/s	2.9
U	8	n/s	3.2
U	9	n/s	3.2
Note: n/s = Not sampled (see Figure 2-2).			

Table 2-6. TCLP Lead Summary and Test Statistics for the Inactive Pottery Factory Site				
Untreated Mean (mg/L)	Treated Mean (mg/L)	Percent Reduction	Treated 95% UCL (mg/L)	Probability That the Actual Treated Mean Is >7.5 mg/L (Students t-test)
403	3.3	99%	3.484	<0.005

2.4.2 Evaluation of P2

Determine whether the portion of total lead in soil that is “bioaccessible,” as measured by an experimental method, can be reduced by at least 25 percent.

The objective was evaluated by collecting samples of untreated and treated soil from the trailer park for soil lead bioaccessibility and analyzing the samples by the SBRC’s SIVM. Table 2-8 presents the results of the SIVM analysis of the untreated and treated soil samples. Soil lead bioaccessibility is the ratio of the amounts of lead that is solubilized during the extraction to the total amount of lead in the soil sample. The concentrations of bioaccessible lead in the untreated soils (mg/kg) are calculated on the basis of total lead measured in the extract and the mass of the soil extracted during the test. The concentrations then are divided by the total concentration of lead measured in the untreated soil to arrive at the percentage of bioaccessible lead in the untreated soils. Identical measurements and calculations are used to calculate the percentage of bioaccessible lead in the treated soils.

Data analysis for the objective consisted of performance of an assessment of data distribution and a parametric test (t-test). An assessment of the results of the validity of the parametric test was performed by the conduct of a distribution-free test (bootstrap analysis).

Table 2-7. TCLP Lead Results for the Trailer Park Site			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
G	Comp	13.2	1.3
L	Comp	11.9	1.4
T	Comp	<0.50	<0.50
Note: Comp = Composite of five sampling locations within an experimental unit (see Figure 2-1).			

Table 2-8. Soil Lead Bioaccessibility Results							
	Untreated Results			Treated Results			Summary
Unit	Total Lead (mg/kg)	Bioaccessible Lead (mg/kg)	Percentage Lead	Total Lead (mg/kg)	Bioaccessible Lead (mg/kg)	Percentage Lead	Percent Reduction
C	645.97	325.29	50.4%	587.41	259.79	44.2%	12.2%
G	6446.73	4536.05	70.4%	8751.09	6025.50	68.9%	2.1%
K	2394.56	1378.70	57.6%	2525.71	1617.51	64.1%	-11.2%
L	7775.47	5209.88	67.0%	7255.24	4780.36	65.9%	1.7%
M	2941.40	1714.58	58.3%	2862.71	1807.93	63.2%	-8.3%
N	2303.51	1338.74	58.1%	1680.93	953.83	56.8%	2.4%
O	2378.06	1140.08	47.9%	2980.51	1553.04	52.1%	-8.7%
Q	726.82	381.79	52.5%	824.93	344.46	41.8%	20.5%
R	1406.92	649.48	46.2%	1397.99	699.90	50.1%	-8.5%
T	339.34	148.68	43.8%	348.95	113.34	32.5%	25.9%

The assessment of data distribution suggested that the soil lead bioaccessibility data followed a normal distribution (for both untreated and treated soils). Therefore, the standard t-test formula for testing for a 100 (1-r0)% reduction in the arithmetic mean was used, with r0 equal to 0.25. Table 2-9 presents a summary of the parametric test statistics, which can be used to determine whether a reduction of at least 25 percent in the soil lead bioaccessibility has been achieved. To conclude that reduction of at least 25 percent has occurred at a significance level of alpha 0.05, the observed t-score should be less than -1.812. On the basis of that criterion, the percent reduction achieved appears to be less than 25 percent.

An assessment of the validity of the results of the parametric test was performed through the conduct of a bootstrap analysis of the sample values. For the bootstrap analysis, samples of size 10 were drawn with replacement 1,000 times from the Soil Rescue soil lead bioaccessibility data. Table 2-10 summarizes the results of that analysis.

The calculated percent reduction in soil lead bioaccessibility was 2.92 percent, with a calculated standard deviation of 3.99 percent and a 95 percent confidence interval of -4.8 percent to 11.2 percent. None of the 1,000 bootstrap calculations were found to exceed a percent reduction value of 25 percent. Therefore, the results of the bootstrap analysis support the results of the parametric test, which

indicate that Soil Rescue did not appear to achieve the goal of at least 25 percent reduction in soil lead bioaccessibility in soils from the trailer park.

2.4.3 Evaluation of Objective S1

Demonstrate the long-term chemical stability of the treated soil.

Various analytical procedures that are indicative of long-term chemical stability were selected for use in evaluating S1. For the demonstration, the long-term chemical stability of the treated soil was evaluated by comparing the analytical results for the untreated soil samples with those for the treated soil samples, using leaching procedures, lead speciation methods, and other inorganic chemical procedures, including the MEP, lead speciation by scanning electron microscopy, lead speciation by the sequential soil serial extraction procedure, Eh, pH, cation exchange capacity, acid neutralization capacity, total lead in soil (as determined by two methods), leachable lead by the SPLP, total phosphates, and leachable phosphates. The discussions below describe the analytical methods, how the methods were used to indicate long-term chemical stability, and the analytical results for each method.

MEP

The MEP was designed to simulate both the initial and subsequent leaching that a waste would undergo in a

Table 2-9. Parametric Test Statistics, Soil Lead Bioaccessibility Data	
Statistic	Data
Value of C_R^1	12.53%
Standard deviation	7.2
t-score ($H_0: C_R$ greater than or equal to 0)	5.499
Level of significance	0.9999
Note: $^1 C_R = C_t - C_u (1 - r_o)$ (see Section 2.3.2.2)	

sanitary landfill. The criterion established for determining whether the results of the MEP demonstrate achievements of S1 (long-term chemical stability) required that the concentrations of lead leached from the treated samples were less than 5.0 mg/L. The criterion is a nominal concentration that would be expected to meet or exceed cleanup goals at some sites; therefore, it is not provided in any federal laws or regulations. Although the MEP was not designed for use on untreated soils, the demonstration plan included analysis of untreated soils using the MEP to provide a basis of comparison with the test results on the treated soils.

Table 2-11 lists the analytical results for the MEP. The data on untreated soil from experimental unit G at the trailer park indicated that the analytical results for the MEP exceeded 5.0 mg/L for days 5 and 6 of the 11-day extraction period. The data on treated soil from the trailer park indicated that the MEP analytical results were consistently less than 5.0 mg/L for the extraction period. Figure 2-3 shows the MEP results for the sample of untreated soil from unit G that were higher than or equal to 5.0 mg/L with the corresponding results for treated soils.

For the five sampling locations at the inactive pottery factory, results for samples of untreated soil were higher than or equal to 5.0 mg/L. The data on treated soil from the inactive pottery factory indicated that the analytical results for the MEP were consistently less than 5.0 mg/L for the extraction period. Figures 2-4 through 2-8 show the results for the samples of untreated soil from the inactive pottery factory that were higher than or equal to 5.0 mg/L, with the corresponding results for treated soil.

On days 7 or 8, the extractions are repeated until concentrations decrease, or until Day 12. Results for Days 10 to 12 were not recorded if there was no increase in lead concentrations from Days 7 or 8 to Day 9.

Table 2-10. Bootstrap Statistical Results for Bioavailable Lead Difference Data	
Statistic	Data
Mean	2.92%
Standard deviation	3.99%
95% confidence interval	(-4.8%, 11.2%)
Number of percent reduction samples > 25%	0/1,000

The analytical results for the MEP indicate that the lead did not leach from the soil treated with Soil Rescue under repetitive precipitation of acid rain conditions. Therefore, the long-term chemical stability of the treated soil, as measured by the MEP, appears to have been enhanced by the addition of Soil Rescue.

Lead Speciation by Scanning Electron Microscopy

This procedure used an EMP technique to determine the frequency of occurrence of 18 lead-bearing phases in soil samples from the trailer park location only. For the demonstration, the mean of the percent frequency of each lead phase was evaluated with regard to the effect the change in that phase will have on the long-term chemical stability of the treated soil. The long-term chemical stability of a soil is enhanced if the application of Soil Rescue increased the frequency of the phases having low solubilities (such as the lead phosphate phase) and decreased the frequency of the species that are highly soluble (such as the lead metal oxide phase). Because of the volume of data generated from the procedure (10 samples for each of 18 metal-bearing phases), the mean of the percent frequency of each phase was determined to compare the analytical results for untreated and treated soils. The unpublished TER provides a table of the raw lead speciation data. The TER is available upon request from the EPA work assignment manager (see Section 1.4 for contact information).

Table 2-12 shows the mean percent frequency of each metal phase for untreated and treated soils, as well as other descriptive statistics. The data suggest that there were potentially significant changes from untreated to treated soils for only 5 of the 18 phases that were evaluated. The frequency of the lead phosphate phase, and possibly the glass phase, increased between the values for untreated and treated soils, a condition that would be indicative of an

Table 2-11. MEP Analytical Results															
Experimental Unit	Untreated/ Treated	Initial Extract (mg/L)	Day 1 (mg/L)	Day 2 (mg/L)	Day 3 (mg/L)	Day 4 (mg/L)	Day 5 (mg/L)	Day 6 (mg/L)	Day 7 (mg/L)	Day 8 (mg/L)	Day 9 (mg/L)	Day 10 ¹ (mg/L)	Day 11 (mg/L)	Day 12 (mg/L)	
Trailer Park															
C	Untreated	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	0.095	0.064	0.087	<0.050				
C	Treated	0.21	0.12	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050				
G	Untreated	1.8	0.38	0.11	0.15	0.057	20	7.4	3.9	2.3	3.3	3.9	2.8		
G	Treated	0.61	0.62	0.98	0.52	0.24	0.078	0.22	0.27	0.34	0.14				
K	Untreated	0.18	0.11	0.14	0.067	<0.050	0.64	1.7	0.62	1.3	0.49				
K	Treated	0.97	0.4	0.33	0.21	0.065	0.057	0.25	0.33	0.33	0.32				
L	Untreated	0.55	0.19	0.25	0.21	0.12	0.12	0.072	0.11	0.11	<0.050				
L	Treated	0.65	0.81	0.58	0.38	0.15	0.076	<0.050	0.06	0.12	0.12	0.29	0.6	0.22	
M	Untreated	1.3	<0.050	0.22	0.11	0.063	0.12	<0.050	<0.050	0.38	0.056				
M	Treated	0.61	0.37	0.48	0.26	0.1	<0.050	<0.050	<0.050	0.06	<0.050				
N	Untreated	0.11	0.12	0.12	<0.050	<0.050	0.088	0.2	0.11	0.6	0.099				
N	Treated	0.91	0.43	0.25	0.11	0.072	<0.050	0.06	<0.050	0.061	0.06				
O	Untreated	0.16	0.075	0.11	<0.050	<0.050	<0.050	<0.050	<0.050	0.3	<0.050				
O	Treated	0.2	0.13	0.087	0.23	0.091	0.1	0.11	0.92	0.059	<0.050				
Q	Untreated	<0.050	0.062	<0.050	<0.050	<0.050	0.077	0.21	0.075	0.22	<0.050				
Q	Treated	0.09	0.061	0.071	0.064	<0.050	<0.050	<0.050	1.5	<0.050	<0.050				
Q (Duplicate)	Untreated	0.075	<0.050	<0.050	<0.050	<0.050	0.28	0.36	<0.050	0.28	0.09				
Q (Duplicate)	Treated	0.078	0.081	<0.050	0.061	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050				
R	Untreated	0.1	0.09	0.086	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	0.094	0.22	0.14		
R	Treated	0.39	0.2	0.15	0.057	<0.050	0.057	0.058	0.073	0.095	0.092				
Note: ¹ After the initial daily extract, nine extractions are performed on each of the following nine days; if the lead concentration is higher in Day 9 than the concentrations in Days 7 or 8, the extractions are repeated until concentrations decrease, or until Day 12. Results for Days 10 to 12 were not recorded if there was no increase in lead concentrations from Days 7 or 8 to Day 9.															
(continued)															

Table 2-11. MEP Analytical Results (continued)													
Experimental Unit	Untreated/ Treated	Initial Extract (mg/L)	Day 1 (mg/L)	Day 2 (mg/L)	Day 3 (mg/L)	Day 4 (mg/L)	Day 5 (mg/L)	Day 6 (mg/L)	Day 7 (mg/L)	Day 8 (mg/L)	Day 9 (mg/L)	Day 10 ¹ (mg/L)	Day 11 (mg/L)
Trailer Park													
T	Untreated	<0.050	<0.050	0.051	<0.050	0.11	0.14	0.45	0.26	0.33	<0.050		
T	Treated	<0.050	<0.050	0.1	<0.050	<0.050	<0.050	<0.050	1.1	<0.050	<0.050		
Inactive Pottery Factory													
U Location 1	Untreated	640	280	120	21	0.14	0.097	0.13	0.21	0.64	0.89	5.5	0.67
U Location 1	Treated	1.1	1.3	2.3	2.4	3.3	2.4	0.99	0.86	0.87	0.72		
U Location 2	Untreated	450	150	57	7.6	0.18	0.24	0.11	0.13	0.097	0.077		
U Location 2	Treated	1	2.2	2.1	2	2.7	2.2	0.87	0.81	0.8	0.12	0.58	
U Location 3	Untreated	260	3.7	0.08	0.31	0.39	0.16	0.15	0.29	0.18	0.14		
U Location 3	Treated	1.1	1.9	2.2	3.5	3.5	2.1	1.1	1	0.9	0.65		
U Location 4	Untreated	203	7.1	0.61	0.26	0.45	0.15	0.12	0.2	0.21	0.18		
U Location 4	Treated	1.1	2.9	2.2	2.1	3.8	2.9	0.88	0.85	0.81	0.69		
U Location 5	Untreated	290	78	2.6	0.39	0.52	0.66	1.7	2.9	2.2	0.84		
U Location 5	Treated	1	1.6	2.5	3.2	3.5	2.8	0.94	0.8	1.1	1.2	0.51	
U Location 6	Treated	1.2	0.5	1.5	1.7	2.8	2	0.68	0.79	0.71	0.55		
U Location 7	Treated	0.97	2.1	1.9	2.5	2.6	2.5	0.92	0.72	0.81	0.82	0.53	
U Location 8	Treated	1	1.8	1.8	1.9	2.8	2.3	0.88	0.64	0.93	0.8	0.36	
U Location 9	Treated	1.2	1.5	1.9	2.7	5.1	3.3	0.72	0.9	1.6	1.1	0.64	
Note: ¹ After the initial daily extract, nine extractions are performed on each of the following nine days; if the lead concentration is higher in Day 9 than the concentrations in Days 7 or 8, the extractions are repeated until concentrations decrease, or until Day 12. Results for Days 10 to 12 were not recorded if there was no increase in lead concentrations from Days 7 or 8 to Day 9.													

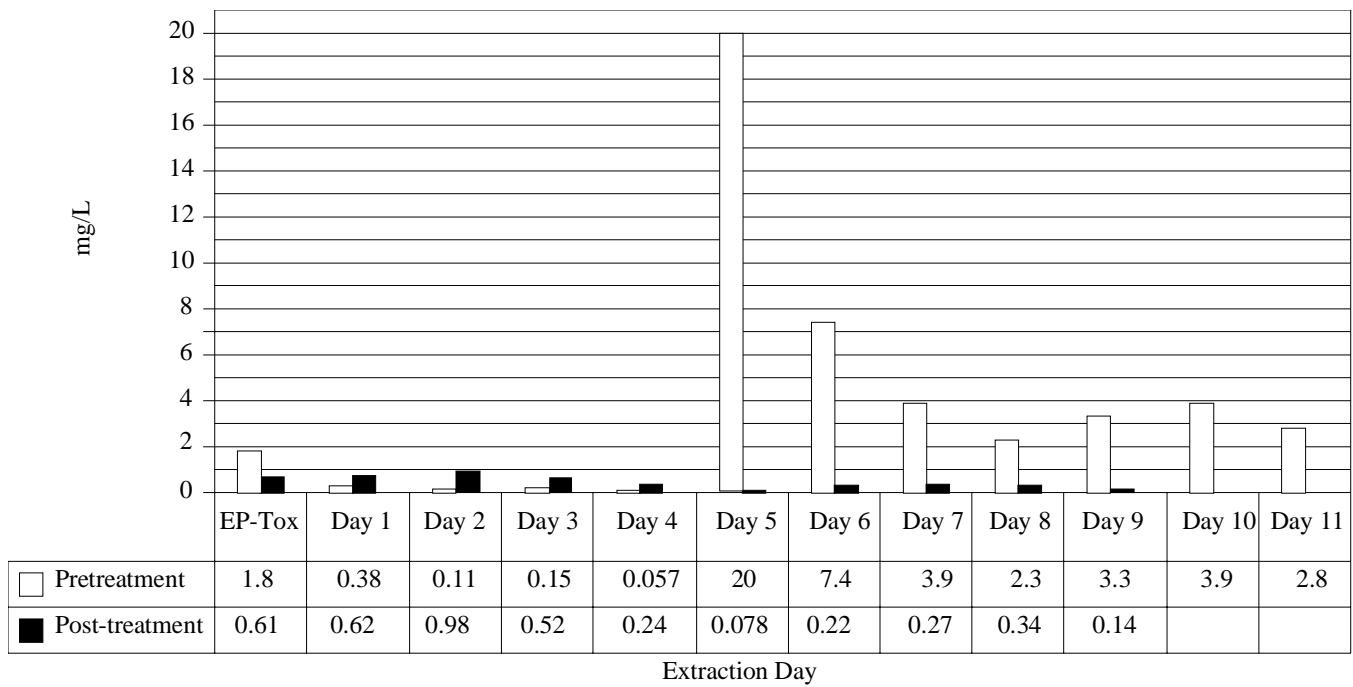


Figure 2-3. MEP lead results for experimental unit G at the trailer park.

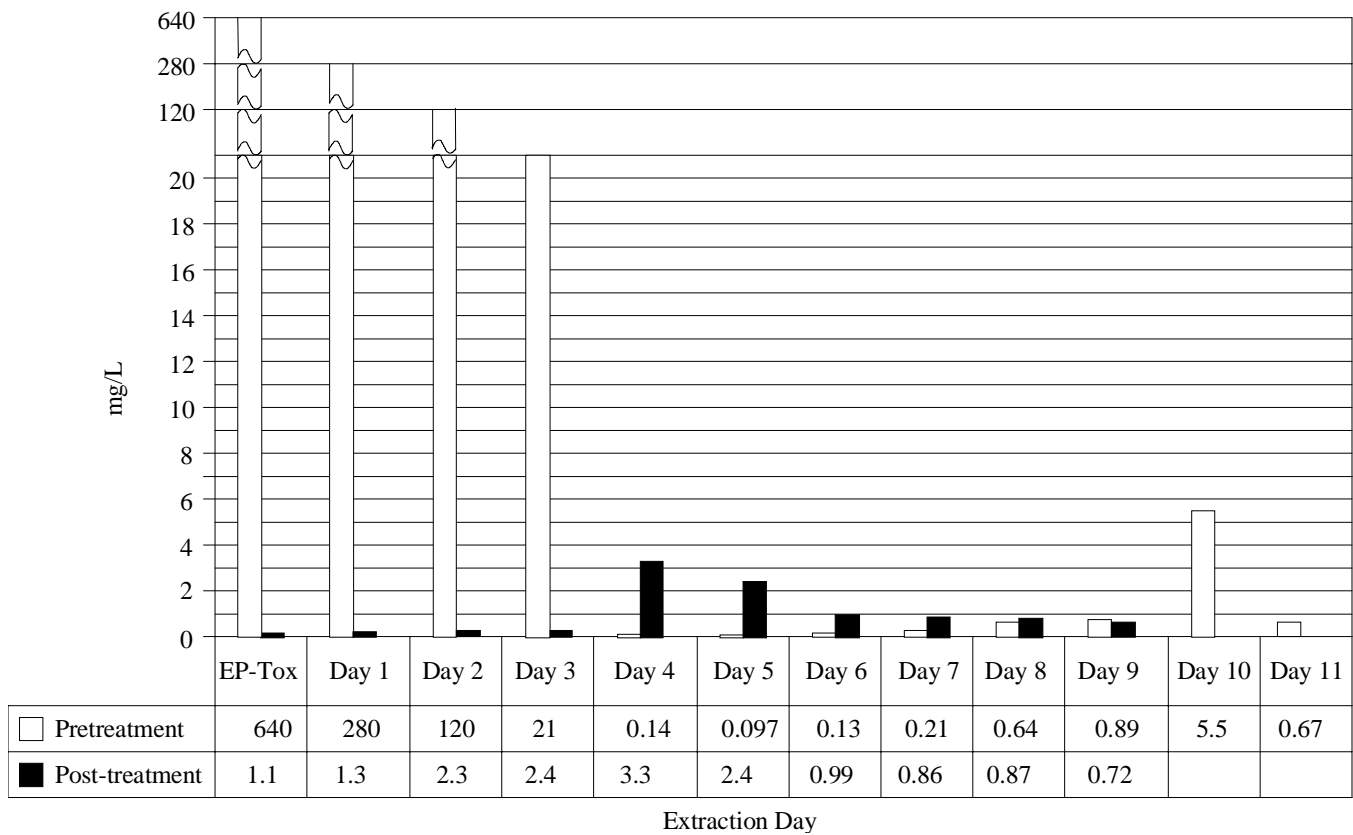


Figure 2-4. MEP lead results for sampling location 1 at the inactive pottery factory.

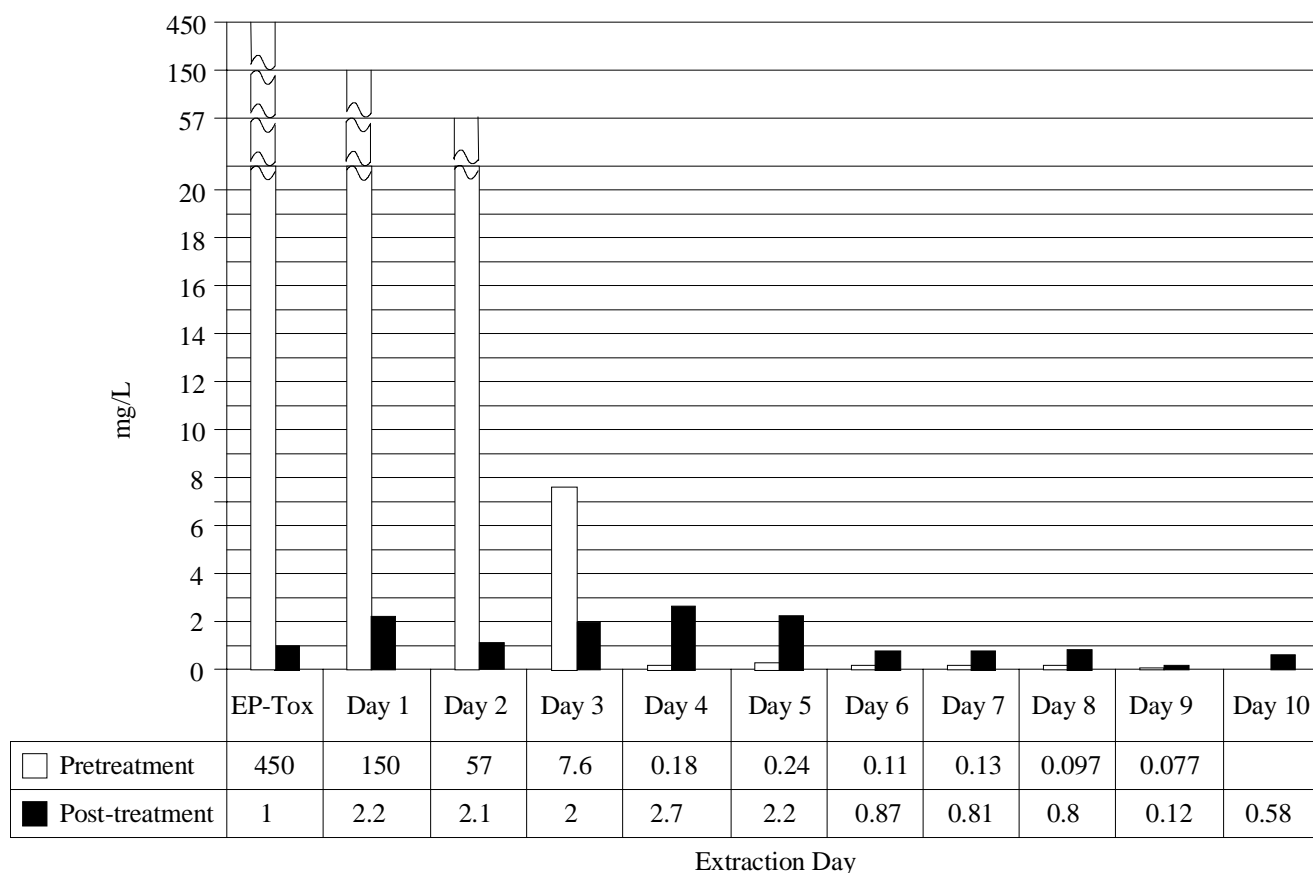


Figure 2-5. MEP lead results for sampling location 2 at the inactive pottery factory.

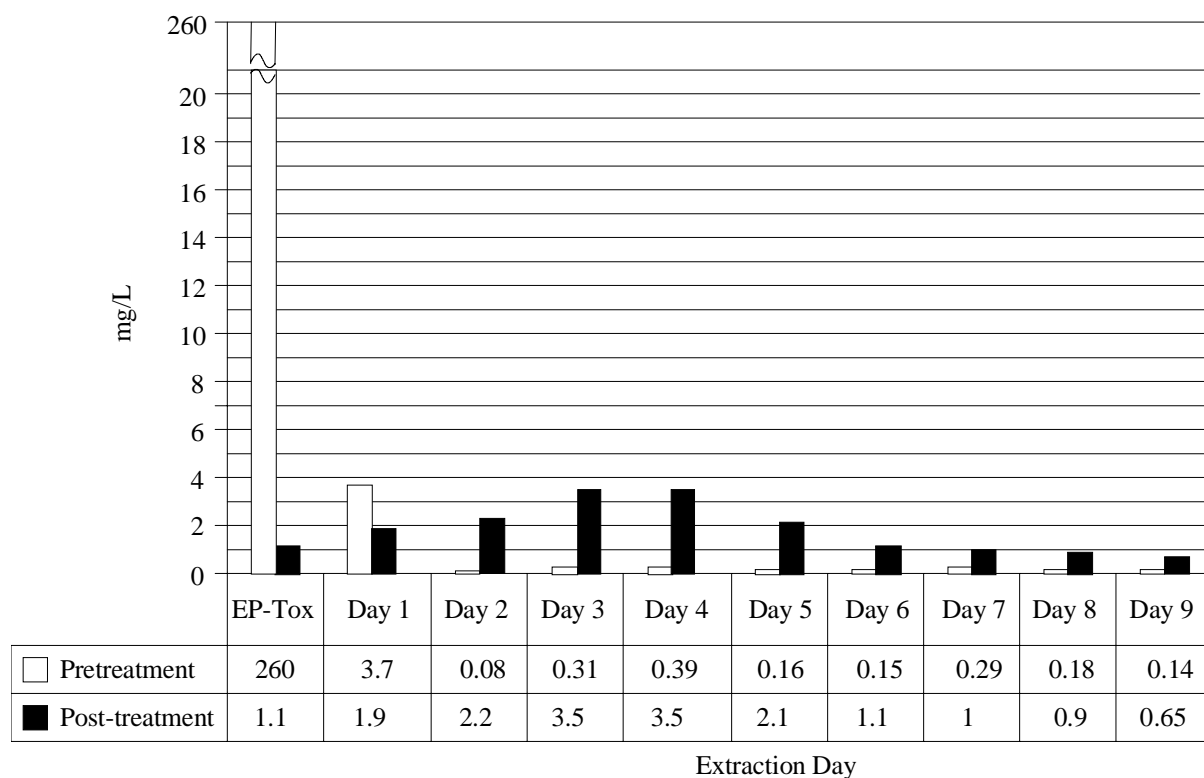


Figure 2-6. MEP lead results for sampling location 3 at the inactive pottery factory.

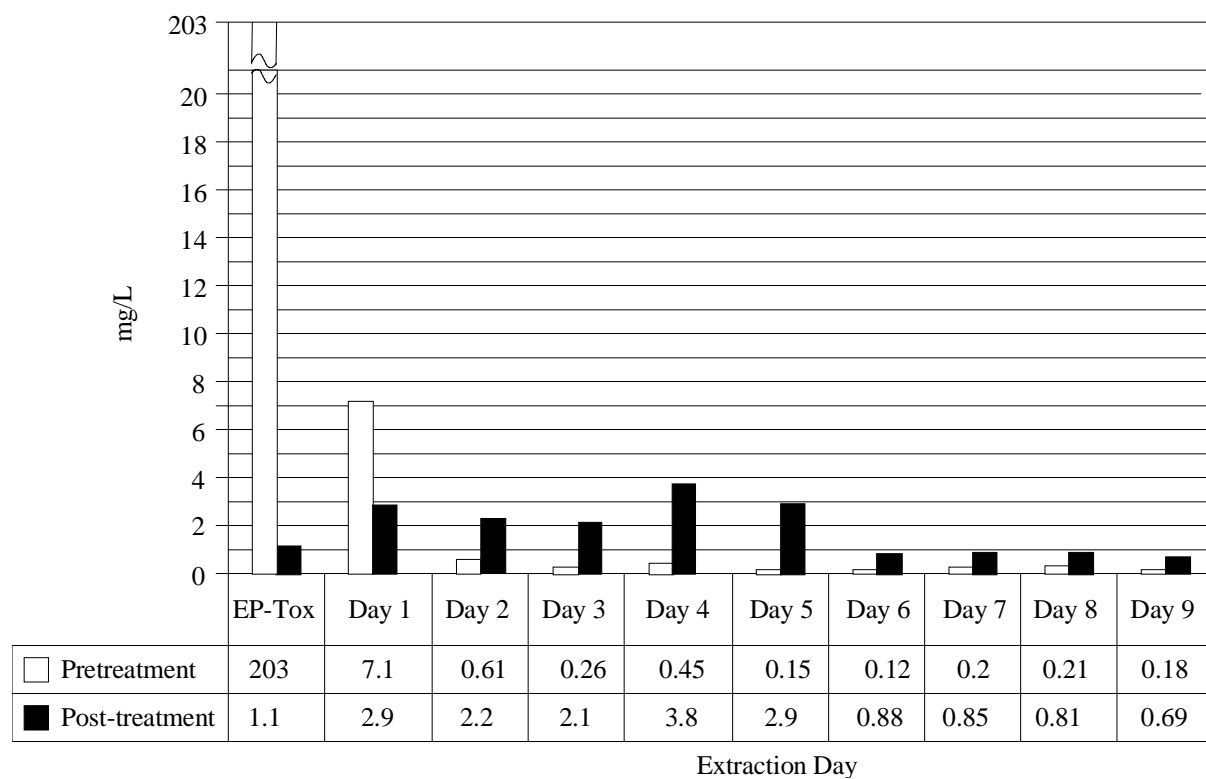


Figure 2-7. MEP lead results for sampling location 4 at the inactive pottery factory.

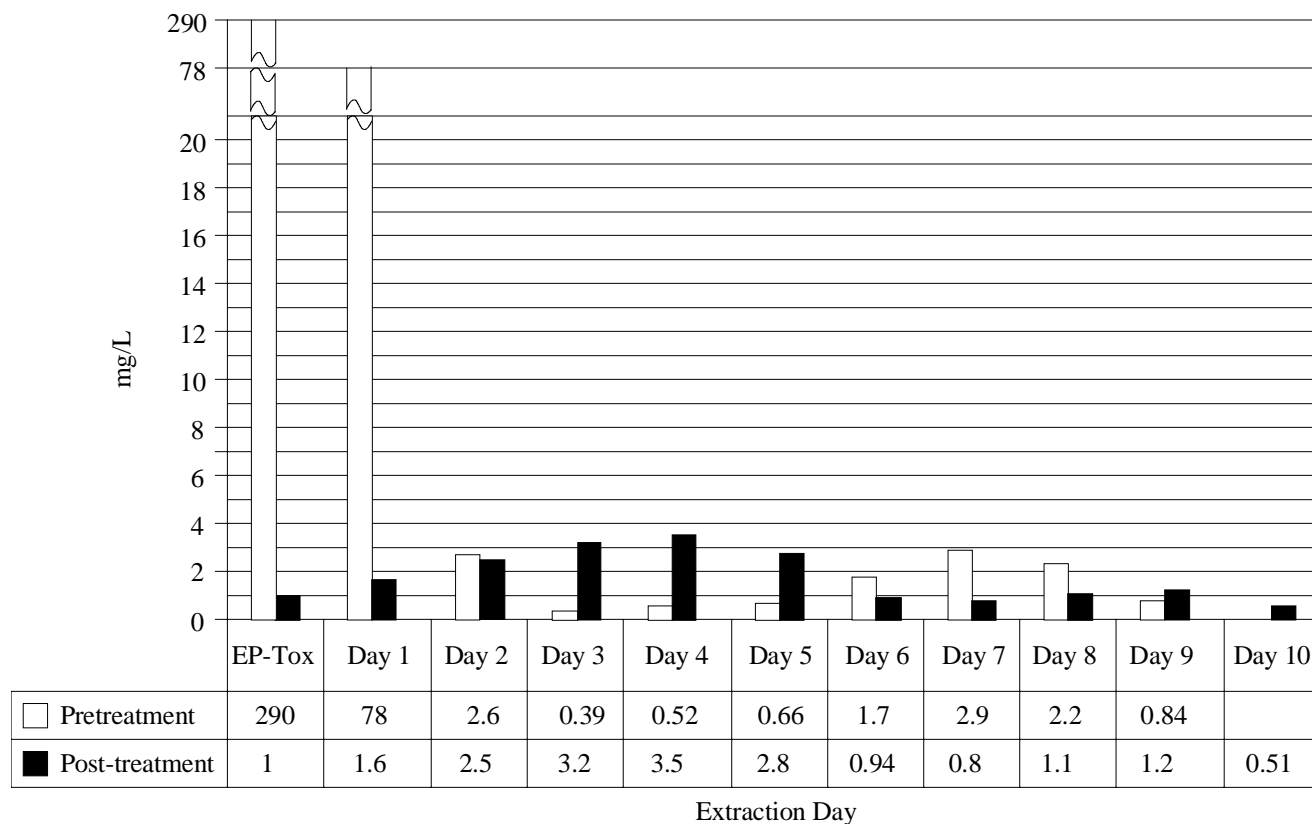


Figure 2-8. MEP lead results for sampling location 5 at the inactive pottery factory.

Table 2-12. Summary of Percent Frequency of Lead Phases Statistical Data						
Phase of Lead	Untreated			Treated		
	Mean	Standard Deviation	Number of Zero Values	Mean	Standard Deviation	Number of Zero Values
Anglesite	0.02	nc ¹	9	0.01	nc	9
Barite	0.1	nc	8	1	nc	6
Brass	0	nc	10	0.13	nc	8
Cerussite	0.41	1.2	8	0.67	1.7	4
Clay	0	nc	10	0	nc	10
Fe-Oxide ²	44.77	15.09	0	21.09	14.47	0
Fe-Pb Sulfate	0.17	nc	7	0	nc	10
Galena	0	nc	10	0	nc	10
Glass ²	39.11	16.15	0	52.52	20.55	0
Mn-Oxide ²	8.39	11.25	3	2.46	5.63	7
Organic ²	1.88	4.55	7	12.23	16.36	3
Pb Vanadate	0	nc	10	0.01	nc	9
PbMO	1.93	1.08	0	1.52	1.07	0
PbSiO ₂	0.58	1.37	6	1.51	2.64	3
Phosphate ²	0.09	0.19	8	5.2	4.58	1
Si-Phosphate	0	nc	10	0.07	nc	9
Slag	2.28	nc	5	1.86	nc	6
Solder	0.02	nc	9	0.04	nc	8
¹ nc = not calculated. Standard deviations were not calculated for data on lead phases that were associated with five or more zero-value data points for both the untreated and treated soils. ² Appears to be a significant difference between treated and untreated soils.						

increase in the long-term chemical stability of the soil. Also indicative of chemical stability are the apparent reductions in the iron oxide and manganese oxide phases of lead. The results also indicate that there was an increase in the organic lead phase, which indicates a reduction in stability from the untreated to the treated soils. Application of Soil Rescue appears to increase the organic lead phase; however, it also appears to increase the less-soluble phosphate phase and reduce the soluble oxide phases of lead in the treated soil. Because of the nature of the speciation test, it is not possible to identify the net result of the changes in the frequencies of those five phases. Therefore, the lead speciation results were not unanimously consistent with the attainment of objective S1; however, it appears that those results suggest that Soil Rescue can enhance the long-term stability of treated soil.

Lead Speciation by Sequential Extraction

This procedure uses sequential chemical extractions with different reagents to determine the concentration of lead that partitions into each of several discrete metal phases. The phases include exchangeable lead, lead bound to carbonates, lead bound to iron oxide, lead bound to manganese oxide, lead bound to organic matter, and residual lead.

The lead in the exchangeable phase, carbonates phase, iron oxide phase, manganese oxide phase, and organic matter phase is subject to release to the environment in a soluble form because of such changes in soil conditions as pH and Eh. The residual phase contains principally primary and secondary minerals that may hold the lead within their crystal structures. Therefore, long-term stability was evaluated by comparing the concentrations of lead in each

phase of the untreated samples with the concentrations of lead in each phase of the treated samples. Long-term stability would be suggested if there are decreases in the concentrations of lead in the exchangeable phase, carbonates phase, iron oxide phase, manganese oxide phase, and organic matter phase, with an increase in the residual phase.

Tables 2-13 and Table 2-14 present the results of the sequential extractions on soil samples from the trailer park and the inactive pottery factory, respectively. On the basis of an assessment of graphical data distribution, the sequential extraction data appear to be distributed normally. Therefore, the data on untreated soils from the trailer park and the inactive pottery factory were analyzed separately through application of a series of individual t-tests extraction.

Table 2-15 displays the summary statistics associated with the sequential extraction data from both locations. Those statistics include the estimated means for the untreated and treated soils, the calculated percent change in those means, and the level of significance of each t-score. Note that, because a total of six simultaneous t-tests were performed, a Bonferroni correction was used to preserve the overall Type 1 error rate. Therefore, no t-score should be considered statistically significant at the 0.05 level unless the corresponding level of significance is less than $0.05/6 = 0.0083$.

As Table 2-15 shows, the results of the sequential serial soil extractions indicate reductions in the concentrations of four of the six lead phases (exchangeable, carbonate, manganese oxide, and iron oxide) and increases in the other two lead phases (organic matter and residual) in soils from both sites. Those results are consistent with those obtained for lead speciation by the SEM procedure (presented in the previous section).

Of the results for the 12 Student t-tests, 8 appear to be statistically significant. The four other results were almost statistically significant; therefore, the changes in the treated soils that these other four tests indicated probably occurred. The four results that were not found to be significant at the 0.05 level of significance include increases in exchangeable and organic matter phases at the trailer park and increases in residual concentrations at both locations. There are significant decreases in the mean concentrations of lead bound to carbonates and lead bound to iron and manganese oxide phases at both sites. Soil from the trailer park also exhibited a significant decrease in lead bound to the exchangeable phase. Soil from the inactive pottery factory

exhibited a significant increase in the organic matter phase.

The results of the statistical analysis indicate that Soil Rescue increased the mean concentrations of the residual phases of lead at both site locations; however, such increases do not appear to be significant at the 0.05 level of significance. Those results also indicate that the application of Soil Rescue significantly reduced the concentrations of three soluble lead phases (carbonate, manganese oxide, and iron oxide) at both sites, with significant and almost-significant reductions of another highly soluble lead phase (exchangeable). Finally, the data indicate that significant and almost-significant increases of another soluble lead phase (organic matter) occurred at both locations. Therefore, the lead speciation results were not unanimously consistent with the attainment of objective S1.

Eh

Eh was evaluated to determine whether the treated soil exhibits an oxidizing or reducing environment. Reducing conditions favor retention of lead in the soil, which may increase the long-term stability of the treated soil. The long-term stability of the treated soil was evaluated by comparing the Eh values for untreated soil with the values for treated soils and by determining whether the soil exhibited an oxidizing or reducing environment. A decrease in the Eh values would suggest long-term stability of the treated soil.

Table 2-16 presents the Eh data for untreated and treated soil from the trailer park, and Table 2-17 presents the Eh data for untreated and treated soil from the inactive pottery factory. These Eh data appear to be normally distributed, based on a graphical data distribution assessment.

Table 2-18 presents the summary statistics associated with the analysis. Included in that table are the observed Eh means for untreated and treated soils, the estimated mean differences, and the levels of significance of the corresponding t-scores for the soil from the trailer park. The increase in the Eh mean level from the untreated to the treated soil appears to be statistically significant. The Eh results from the trailer park therefore indicate that the application of Soil Rescue has increased the Eh of the soil, which does not indicate long-term stability of the soil treated with Soil Rescue at the trailer park. For the soil from the inactive pottery factory, the decrease in the Eh mean from the untreated to the treated soil appears not to be significant and therefore would not indicate long-term

Table 2-13. Sequential Serial Soil Extracts Results, Trailer Park

Unit	Sampling Location	Untreated						Treated					
		1	2	3	4	5	6	1	2	3	4	5	6
C	Comp	3.46	2.65	1.657	6.19	7.29	187	0.13	9.35	0.103	2.27	9.99	381
G	Comp	80.4	81.42	23.25	62.5	61.75	2026	3.62	22.72	8.935	46.62	245.5	4,064
K	Comp	37.5	36.66	6.985	30.22	15.44	781	3.03	7.92	5.131	20.64	52.23	2,583
L	Comp	60.9	105.4	9.039	88.69	42.98	2386	15.14	57.48	3.949	81.28	125.1	3,903
M	Comp	11	70.49	15.97	32.64	14.96	543	6.53	18.37	12.4	31.92	33.23	790
N	Comp	13.2	18.57	11.93	23.76	22.06	504	0.7	3.06	1.331	13.03	36.29	799
O	Comp	2.86	36.3	3.897	29.59	19.47	516	0.62	0.85	2.094	20.47	48.96	1,371
Q	Comp	12.4	8.08	3.724	5.68	7.18	1889	0.46	1.59	0.125	3.33	16.72	551
R	Comp	20	12.22	5.485	14.22	9.494	325	1.04	4.49	1.006	9.22	24.63	786
T	Comp	5.55	2.25	1.127	4.89	3.24	71	0.11	0.43	0.1	0.84	5.76	220

Note: 1 = Exchangeable phase (mg/L Pb), 2 = Carbonate phase (mg/L Pb), 3 = Manganese oxide phase (mg/L Pb), 4 = Iron oxide phase (mg/L Pb), 5 = Organic matter phase (mg/L Pb), 6 = Residual phases (mg/L Pb).

Table 2-14. Sequential Serial Soil Extracts Results, Inactive Pottery Factory

Unit	Sampling Location	Untreated						Treated					
		1	2	3	4	5	6	1	2	3	4	5	6
U	1	133.7	1,506	230.3	515.7	255.2	14,446	22.99	255.7	13.83	194.8	1,326	15,749
U	2	117.7	1,349	142.7	579.9	230.3	13,491	22.1	188.5	6.39	213.2	1,329	18,054
U	3	180.4	2,213	261.3	766.9	215.5	13,600	30.18	255.6	17.92	159.6	1,662	23,739
U	4	141.6	1,506	183.4	600.8	285	13,328	32.17	332.4	22.42	198.7	1,579	18,002
U	5	120.3	1,078	195.9	663.9	240.7	13,872	34.59	156.9	16.99	226.5	1,348	17,223
U	6	n/s	n/s	n/s	n/s	n/s	n/s	23.82	304.10	18.24	254.00	1,485	18,157
U	7	n/s	n/s	n/s	n/s	n/s	n/s	14.54	183.20	11.52	213.60	1,234	16,384
U	8	n/s	n/s	n/s	n/s	n/s	n/s	30.89	186.70	8.21	192.30	1,107	15,216
U	9	n/s	n/s	n/s	n/s	n/s	n/s	44.26	233.60	56.17	175.80	1,294	10,843

Note: 1 = Exchangeable phase (mg/L Pb); 2 = Carbonate phase (mg/L Pb); 3 = Manganese oxide phase (mg/L Pb); 4 = Iron oxide phase (mg/L Pb), 5 = Organic matter phase (mg/L Pb); 6 = Residual phases (mg/L Pb); n/s = not sampled.

Table 2-15. Sequential Serial Soil Extracts: Summary Statistics				
Phase	Untreated Mean (mg/L Pb)	Treated Mean (mg/L Pb)	Mean Difference (Untreated - Treated)	Significance level
Trailer Park				
Exchangeable	24.73	3.14	21.59	0.009
Carbonate	37.41	12.63	24.78	0.004 ¹
Manganese Oxide	8.31	3.5	4.81	0.003 ¹
Iron Oxide	29.84	22.96	6.88	0.0005 ¹
Organic Matter	20.39	59.86	-39.47	0.03
Residual	922.8	1,545	-622.2	0.02
Inactive Pottery Factory				
Exchangeable	138.73	28.39	110.34	0.0002 ¹
Carbonate	1,530.45	232.99	1,297.46	0.001 ¹
Manganese Oxide	202.72	19.08	183.64	0.0002 ¹
Iron Oxide	625.44	203.18	422.26	0.0002 ¹
Organic Matter	385.6	1,373.9	-988.3	0.00000001 ¹
Residual	13,751	17,040.78	-3,289.78	0.009
Notes: Hypothesis associated with significance level is H_0 : mean untreated - mean treated = 0. ¹ Significant difference between treated and untreated soil (A significance level of 0.0083 or lower is needed to declare a significant difference, based on a Bonferroni correction needed to preserve the significance level of 0.05).				

Table 2-16. Trailer Park Eh Analytical Results			
Experimental Unit	Sampling Location	Untreated Eh (mV)	Treated Eh (mV)
C	Composite	620	590
G	Composite	690	580
K	Composite	620	530
L	Composite	570	770
M	Composite	490	1,100
N	Composite	600	700
O	Composite	570	800
Q	Composite	500	810
R	Composite	550	820
T	Composite	570	670

Table 2-17. Inactive Pottery Factory Eh Analytical Results			
Experimental Unit	Sampling Location	Untreated Eh (mV)	Treated Eh (mV)
U	1	530	530
U	2	890	610
U	3	590	560
U	4	650	570
U	5	550	530
U	6	n/s	540
U	7	n/s	540
U	8	n/s	580
U	9	n/s	570
Note: n/s = not sampled.			

Table 2-18. Eh Summary Statistics		
Statistic	Trailer Park Data (mV)	Inactive Pottery Factory Data (mV)
Untreated Mean (Standard deviation)	578 (59)	642 (146)
Treated Mean (Standard deviation)	737 (165)	559 (27)
Mean Difference (Untreated - Treated)	159	-83
Significance level	0.02	0.14
Note: Hypothesis associated with significance level is H_0 : mean untreated - mean treated = 0. A paired t-test was conducted on data from the trailer park, and an unpaired t-test assuming unequal variances between treated and untreated samples was conducted on the data from the pottery factory.		

stability. Overall, the results suggest that the application of Soil Rescue may either increase or not significantly affect the Eh of the treated soil ; however, such changes in Eh did not appear to bring along increases in lead-oxide and manganese-oxide phases of lead, as evidenced by the reductions in the phases observed in the data from two lead speciation evaluations (discussed above). In summary, long-term chemical stability was not indicated for soils treated by Soil Rescue by the analytical results from oxidation-reduction (Eh) analysis.

pH

In general, the maximum retention of lead is achieved in soils that are characterized by a pH higher than 7.0, and the solubility of lead is generally lower in soils that have a pH between 7.0 and 10.0. Therefore, the pH values of untreated and treated soils were evaluated to determine whether the pH was higher than 7.0 in the samples of treated soil and to determine whether the pH values had increased after treatment with Soil Rescue.

Table 2-19 presents the analytical results for pH in the soil from the trailer park. Table 2-20 displays the pH analytical results for pH in the soil from the inactive pottery factory. On the basis of an assessment of data distribution, the pH data appear to be distributed normally; however, pH is the negative log of hydrogen ion activity. Therefore, pH data on the untreated and the treated soils were converted to molar concentration units and then were analyzed separately for the trailer park and the inactive pottery factory, through the use of individual t-tests.

Table 2-21 shows the summary statistics associated with the analysis. Included in the table are the observed pH means for untreated and treated soils, the estimated mean differences, and the levels of significance of corresponding t-scores. Note that the increase in pH mean levels from

untreated to treated soils at each site appears to be statistically significant. In addition, 4 of 10 pH values for treated soils from the trailer park are within the optimum range, and all pH values for treated soil from the inactive pottery factory are within the optimum range of 7.0 to 10.0. On the basis of those results, the application of Soil Rescue appears to have enhanced the long-term stability of the treated soil.

Cation Exchange Capacity

The objective of the tests for CEC was to determine if Soil Rescue could increase the CEC, which would indicate an increase in the ability of the soil to prevent migration of lead. The analytical results for CEC from one untreated soil sample were compared with those from one treated soil sample collected at both the trailer park and the inactive pottery factory to determine whether the cations in Soil Rescue changed the mobility of the lead in the soil. Table 2-22 displays the CEC data from the trailer park, and Table 2-23 displays the CEC data from the inactive pottery factory. The CEC data for the trailer park show an increase from the result for untreated soil of 0.12 meq/g to the result for treated soil of 0.22 meq/g. CEC data for the inactive pottery factory also show an increase in the CEC from the result for untreated soil of 0.09 meq/g to the result for treated soil of 0.26 meq/g.

At both sites, the availability of exchangeable potassium showed the largest increase. The total observed increases in the available cations would be expected to reduce the migration rates and the total distances of migration of the total masses of lead in the soils at both sites. Therefore, improvements in the CEC indicate that the application of Soil Rescue appears to have enhanced the long-term stability of the treated soil. However, the results are not quantitative because CEC tests were conducted on only one sample from each site.

Table 2-19. Trailer Park pH Analytical Results			
Experimental Unit	Sampling Location	Untreated	Treated
C	Composite	5.9	6.8
G	Composite	6.2	7.5
K	Composite	5.9	6.5
L	Composite	6.5	6.7
M	Composite	6.9	6.7
N	Composite	6.3	7.8
O	Composite	7.8	6.8
Q	Composite	5.3	7.2
R	Composite	5.3	7.9
T	Composite	4.8	6.6

Table 2-20. Inactive Pottery Factory pH Analytical Results			
Experimental Unit	Sampling Location	Untreated	Treated
U	1	6.9	8.2
U	2	7.5	8.0
U	3	7.4	7.8
U	4	7.5	7.7
U	5	7.4	7.9
U	6	n/s	8.2
U	7	n/s	7.8
U	8	n/s	7.9
U	9	n/s	8.1
Note: n/s = Not sampled			

Table 2-21. pH Summary Statistics		
Statistic	Trailer Park Data	Inactive Pottery Factory Data
Untreated Mean ¹	5.52	7.27
Treated Mean ¹	6.85	7.92
Mean Difference (Untreated - Treated)	1.33	0.65
Significance level	0.041	0.049
Notes: Hypothesis associated with significance level is H_0 : mean untreated - mean treated = 0. A paired t-test was conducted on data from the trailer park, and an unpaired t-test assuming unequal variances between treated and untreated samples was conducted on the data from the pottery factory. ¹ Mean values are reported as pH; however, they were calculated based on molar concentration units obtained by conversion of the individual pH unit measurements shown in tables 2-19 and 2-20.		

Table 2-22. CEC Analytical Results for Soil from the Trailer Park								
Untreated/ Treated	Na (meq/g)	Al (meq/g)	Ca (meq/g)	Mg (meq/g)	K (meq/g)	Fe (meq/g)	Mn (meq/g)	Total (meq/g)
Untreated	0.0022	0.0022	0.0987	0.0129	0.0046	0.0000	0.0003	0.1190
Treated	0.0023	0.0001	0.0544	0.0108	0.1475	0.0000	0.0048	0.2199
Note: meq/g = milliequivalents per gram = weight of element in soil (mg) ÷ (atomic weight [g] ÷ valence) per gram of soil.								

Table 2-23. CEC Analytical Results for Soil from the Inactive Pottery Factory

Untreated/ Treated	Na (meq/g)	Al (meq/g)	Ca (meq/g)	Mg (meq/g)	K (meq/g)	Fe (meq/g)	Mn (meq/g)	Total (meq/g)
Untreated	0.0038	0.0001	0.0759	0.0083	0.0010	0.0000	0.0000	0.0893
Treated	0.0141	0.0001	0.0406	0.0150	0.1880	0.0000	0.0047	0.2626

Note: meq/g = milliequivalents per gram = weight of element in soil (mg) \times \div (atomic weight [g] \div valence) per gram of soil.

Acid Neutralization Capacity

One soil sample was collected before and another after the application of Soil Rescue at the trailer park and the inactive pottery factory; all four samples were analyzed for acid neutralization capacity. Increasing the acid neutralization capacity provides more ligands for formation of the more stable lead complexes, thereby enhancing the long-term stability of treated soil. Data on acid neutralization capacity for soil from the trailer park indicate that there was an increase from the result for untreated soil of 0.0846 meq/g to the result for treated soils of 0.1214 meq/g. The data on acid neutralization capacity data for the inactive pottery factory indicate that there was a decrease from the data on the result for untreated soil of 0.6329 meq/g to the result for treated soil of 0.5013 meq/g. Because the analytical results were not consistent at the two sites, the data do not suggest that the long-term stability of the treated soil was enhanced by the application of Soil Rescue. However, the results are not statistically conclusive because only one pair of soil samples was collected at each location.

Total Lead in Soil

Two analytical procedures were used to determine total concentrations of lead in the soil. One procedure, SW-846 Method 3050B, uses a nitric acid solution to digest the lead. The solution is a very strong acid that dissolves almost all of lead in a sample that could become “environmentally available” (EPA 1996); however, the method is not a total digestion technique. Lead bound in silicates and lead bound to organics may not be dissolved by this method. Therefore, a portion of each soil sample was also digested by hydrofluoric acid. That procedure digests the siliceous and organic matrices and other complex matrices to produce a total concentration of lead.

Both procedures were used to determine whether Soil Rescue forms complex matrices that are not dissolved readily. Binding of the lead into complex matrices should reduce the concentration of lead that is environmentally

available. If the concentration of lead determined by nitric acid digestion decreases after treatment while the concentration of lead determined by hydrofluoric acid digestion does not change significantly, the risk of exposure to environmentally available lead is reduced. If the concentration of lead determined by nitric acid digestion increases after treatment while the concentration of lead determined by hydrofluoric acid digestion does not change significantly, the risk of exposure to environmentally available lead is increased. If the concentration of lead determined by both procedures does not change significantly, the risk of exposure to environmentally available lead is unchanged. However, if the concentration of lead determined by hydrofluoric acid digestion increases significantly, the distribution of lead in complex matrices may follow a non-normal pattern. It should be noted that these tests were extremely aggressive tests, thus meeting the acceptance criteria established for these tests was not as important as meeting the acceptance criteria of other tests involving long-term chemical stability.

Table 2-24 lists the concentrations of lead determined by nitric acid digestion of untreated and treated soil from the trailer park, and Table 2-25 lists the concentrations of lead acid digestion of untreated and treated soil from the inactive pottery factory. The data appear to be distributed normally, as indicated by a graphical assessment of data distribution. Therefore, the differences between total lead in treated and untreated soils were analyzed separately for the trailer park and the inactive pottery factory, through the use of separate Student t-tests.

Table 2-26 displays the summary statistics associated with the analysis. The statistics include the estimated untreated and treated mean concentrations of lead, the calculated percent change in the means, and the levels of significance of the t-scores. The observed mean concentration of lead in soil from the trailer park increased from 1,802.8 mg/kg to 2,168.9 mg/kg, while the mean concentration of lead in soil from the inactive pottery factory decreased from 34,740 mg/kg to 31,422.2 mg/kg. However, the corresponding t-scores indicate that neither of the observed

Table 2-24. Lead Analytical Results for Nitric Acid Digestion for Soil from the Trailer Park			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
C	Composite	345	409
G	Composite	4,330	4,900
K	Composite	2,170	1,580
L	Composite	4,440	9,260
M	Composite	2,200	1,480
N	Composite	1,320	1,090
O	Composite	1,550	1,510
Q	Composite	496	478
R	Composite	907	766
T	Composite	270	216

Table 2-25. Lead Analytical Results for Nitric Acid Digestion for Soil from the Inactive Pottery Factory			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
U	1	40,600	30,900
U	2	28,200	22,400
U	3	41,100	42,700
U	4	36,300	29,500
U	5	27,500	26,800
U	6	n/s	43,300
U	7	n/s	34,200
U	8	n/s	22,300
U	9	n/s	30,700
Note: n/s = not sampled.			

Table 2-26. Summary Statistics for Nitric Acid Digestion		
Statistic	Trailer Park Data (mg/kg)	Inactive Pottery Factory Data (mg/kg)
Untreated mean (Standard deviation)	1,802.8 (1,524)	34,740.0 (6,565)
Treated mean (Standard deviation)	2,168.9 (2,826)	31,422.2 (7,636)
Mean Difference (Untreated - Treated)	-366.1	3,317.80
Level of significance	0.2	0.2
Note: Hypothesis associated with significance level is H_0 : mean untreated - mean treated = 0. A paired t-test was conducted on data from the trailer park, and an unpaired t-test assuming unequal variances between treated and untreated samples was conducted on the data from the pottery factory.		

differences is statistically significant. Therefore, the statistical analysis of the data suggests that, for both sites, there are no significant differences in mean concentrations of total lead between untreated and treated soils using the nitric acid digestion method for total lead.

Table 2-27 presents the concentrations of lead determined by hydrofluoric acid digestion of untreated and treated soil from the trailer park, and Table 2-28 presents the concentrations of lead determined by hydrofluoric acid digestion of untreated and treated soils from for the inactive pottery factory. The data also appear to be distributed normally, and the estimates of sample variance

for the data from both locations again appear to be approximately equivalent. Therefore, separate Student t-tests were performed on the data from the pottery factory and the data from the trailer park to compare the differences in total concentrations of lead in untreated and treated soils.

Table 2-29 displays the summary statistics associated with the analyses. The statistics again include the estimated mean concentrations of lead for untreated and treated soil, the calculated percent change in the means, and the level of significance of the t-scores. The observed mean concentration of lead in soil from the trailer park increased

Table 2-27. Lead Analytical Results Using Hydrofluoric Acid Digestion for the Trailer Park			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
C	Composite	413	398
G	Composite	4,080	13,000
K	Composite	2,010	2,660
L	Composite	6,140	6,420
M	Composite	838	2,740
N	Composite	1,060	1,150
O	Composite	808	1,710
Q	Composite	507	450
R	Composite	825	772
T	Composite	301	275

Table 2-28. Lead Analytical Results Using Hydrofluoric Acid Digestion for the Inactive Pottery Factory			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
U	1	42,900	47,800
U	2	49,100	39,400
U	3	55,700	42,300
U	4	47,000	33,700
U	5	47,800	27,200
U	6	n/s	40,900
U	7	n/s	33,200
U	8	n/s	31,800
U	9	n/s	35,800
Note: n/s = not sampled.			

Table 2-29. Summary Statistics for Hydrofluoric Acid Digestion		
Statistic	Trailer Park Data (mg/kg)	Inactive Pottery Factory Data (mg/kg)
Untreated Mean (Standard deviation)	1,698.2 (1,921)	48,500 (4,645)
Treated Mean (Standard deviation)	2,957.5 (3,981)	36,900 (6,279)
Mean Difference (Untreated - Treated)	-1,259.30	11,600
Significance level	0.092	0.002
Note: Hypothesis associated with significance level is H_0 : mean untreated - mean treated = 0. A paired t-test was conducted on data from the trailer park, and an unpaired t-test assuming unequal variances between treated and untreated samples was conducted on the data from the pottery factory.		

from 1,698.2 mg/kg to 2957.5 mg/kg, while the mean concentration of lead in soil from the pottery factory decreased from 48,500 mg/kg to 36,900 mg/kg. The change in the mean concentrations of lead is not statistically significant at the trailer park, according to the t-score value, which is the expected outcome of the analysis. However, the decrease in total concentrations of lead at the inactive pottery factory is considered significant. Therefore, the statistical analysis of those data suggests that there was no difference in concentrations of lead between treated and untreated soils for soils from the trailer park and a significant decrease in mean concentration of lead in treated soil from the pottery factory, as determined by the hydrofluoric acid digestion method. The reason for the significant decrease is unknown; however, it is possible that the drop in total lead concentrations (as measured by the hydrofluoric acid digestion method) at the inactive pottery factory may have been the result of the sampling

efforts conducted on the untreated soils, which may have removed some hot spots of high lead concentrations that were bound in stable matrices (therefore, no more of such materials may have remained when the soils were sampled after the application of Soil Rescue).

SPLP Lead

The SPLP concentrations of lead in untreated soil were compared with the SPLP concentrations of lead in treated soil to determine whether the application of Soil Rescue decreased the solubility of the lead in the soil. The criterion selected for determining whether the application of Soil Rescue had an effect on the soil was a concentration of SPLP lead in treated soil of less than 5.0 mg/L.

Table 2-30 lists the concentrations of SPLP lead in untreated and treated soil from the trailer park. The

Table 2-30. SPLP Lead Analytical Results for Soil from the Trailer Park			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
C	Composite	<0.50	<0.50
G	Composite	<0.50	3.1
K	Composite	<0.50	<0.50
L	Composite	<0.50	<0.50
M	Composite	<0.50	1.2
N	Composite	<0.50	<0.50
O	Composite	<0.50	0.67
Q	Composite	<0.50	<0.50
R	Composite	<0.50	<0.50
T	Composite	<0.50	<0.50

concentrations of SPLP lead in untreated soil from the trailer park all were lower than the detection limit of 0.5 mg/L. Of the 10 samples of treated soil from the trailer park, 3 contained concentrations of SPLP lead that were higher than the detection limit, but none of those concentrations exceeded the criterion of 5.0 mg/L. The concentrations of SPLP lead in untreated soil from the trailer park indicate that the contaminated soil would not require treatment.

A parametric statistical analysis of the concentrations of SPLP lead in treated soil cannot be performed because of the excessive number of nondetects. However, the following nonparametric argument can be made to support a conclusion that SPLP mean concentration of SPLP lead in treated soil does not exceed 5.0 mg/L. If the mean was greater than or equal to 5.0 mg/L, the probability of observing an individual concentration of SPLP lead higher than 5.0 mg/L would be at least 0.5. Therefore, the probability of observing 10 independent samples of treated soil at less than 5.0 mg/L could be no more than $(0.5)^{10} = 0.00098$. Therefore, the hypothesis that the mean concentration of SPLP lead in treated soil from the trailer park exceeds 5.0 mg/L is rejected at a 0.001 level of significance. The statistical analysis of untreated and treated soil from the trailer park did not indicate a statistically significant change in concentrations of SPLP lead.

Table 2-31 lists the concentrations of SPLP lead from the inactive pottery factory. The concentrations of SPLP lead in untreated soil from the inactive pottery factory all were lower than the detection limit of 0.5 mg/L. All the

concentrations of SPLP lead in treated soil from the inactive pottery factory exceed the regulatory limit of 5 mg/L. Table 2-32 shows the pertinent summary statistics for SPLP data on treated soil from the inactive pottery factory. The statistics include the estimated mean, standard deviation, and 95 percent upper confidence limit (UCL) for the SPLP data on treated soil, assuming that the data are distributed normally. The estimated mean concentration of SPLP lead in soil from the inactive pottery factory was 8.78 mg/L, with a 95 percent UCL of 9.76 mg/L. Because the UCL estimate is significantly higher than 5.0 mg/L, the concentrations of SPLP lead in the treated soil indicate that the treated soil may leach small amounts of lead. In fact, the mean concentrations of SPLP lead in the treated soils from the inactive pottery factory appear to be significantly higher than the mean concentrations of TCLP lead (3.3 mg/L; see Table 2-6) in those same treated soils. These results are unexpected, since the TCLP generally results in higher concentrations of leachable lead than the SPLP. Those differences cannot be explained without further testing. However, the different acids used for the TCLP and the SPLP (acetic for CLP; sulfuric and nitric for the SPLP) may have contributed to the differences. Further, the results of the MEP tests (in which acetic acid is used initially, followed by sulfuric and nitric acids) that were conducted on soils from the inactive pottery factory and shown in Table 2-11 indicate that the concentrations of lead leached from both untreated and treated soils by sulfuric and nitric acids are much higher than those shown in Table 2-31.

Summary of Results for Objective S1			
Procedure	Results	Interpretation	
		Trailer Park	Pottery Factory
MEP	All results met the acceptance criteria for S1 (see Table 2-4).	Soil Rescue exhibits long-term stability, as indicated by the results of this procedure.	
Lead speciation by SEM	Results for 4 of 18 phases of lead met the acceptance criteria for S1, and results for one phase did not meet the criteria. Results for the other 13 phases did not appear to be affected by the treatment.	Inconclusive: Lead in phosphates and glass appears to increase, and lead in oxide phases appear to decrease after addition of Soil Rescue; however, lead in organic matter appears to increase.	This procedure was not conducted on soils from this location.
Lead speciation by sequential extractions	Results for three of six phases of lead at one site, and four of six at the other site met the acceptance criteria for S1. One phase did not meet the criteria, and the four other phases did not appear to be affected by the treatment.	Inconclusive: Soil Rescue exhibits some long-term stability, as indicated by the results of this procedure. Lead in carbonate and oxide phases was reduced, and exchangeable lead may have been reduced. However, organic lead may have been increased, and residual lead appeared to be unchanged.	Soil Rescue did not increase the long-term stability, as indicated by this procedure. Exchangeable lead and lead in carbonate and oxide phases were reduced, and residual lead may have been increased. However, organic lead increased.
Eh	The criterion for S1 was not met for either site.	Soil Rescue did not increase long-term stability, as indicated by the results of this procedure.	Soil Rescue did not increase long-term stability, as indicated by the results of this procedure.
pH	All results met the acceptance criteria for S1 (see Table 2-4).	Soil Rescue increased long-term stability, as indicated by the results of this procedure.	
CEC ¹	All results met the acceptance criteria for S1 (see Table 2-4).	Soil Rescue increased long-term stability, as indicated by the results of this procedure.	
Acid neutralization capacity ¹	The criterion for S1 was met for one site but was not met for the other site.	Soil Rescue increases long-term stability, as indicated by the results of this procedure.	Soil Rescue did not exhibit long-term stability, based on the results of this procedure.
Total lead by nitric acid digestion compared with total lead by hydrofluoric acid digestion	None of the results met the acceptance criteria for S1 (see Table 2-4).	Soil Rescue does not increase long-term stability, as indicated by the results of this procedure.	
SPLP lead	The acceptance criterion for S1 was met at one site but was not met at the other site.	Soil Rescue increases long-term stability, as indicated by the results of this procedure. However, SPLP lead concentrations were significantly higher in the treated soils.	Soil Rescue did not exhibit long-term stability, based on the results of this procedure.
Total phosphate	None of the results met the acceptance criteria for S1 (see Table 2-4).	Soil Rescue does not increase long-term stability, as indicated by the results of this procedure. However, the increase in concentrations of phosphate in treated soils is related only indirectly to long-term stability and therefore is not as meaningful as the findings for most of the other procedures conducted.	
SPLP phosphate	None of the results met the acceptance criteria for Objective S1 (See Table 2-4).	Soil Rescue does not increase long-term stability, as indicated by the results of this procedure.	
Note: ¹ These tests are considered to be qualitative, because only one sample at each site was tested by this procedure.			

Table 2-31. SPLP Lead Analytical Results for Soil from the Inactive Pottery Factory			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
U	1	<0.50	8.8
U	2	<0.50	7.6
U	3	<0.50	10.7
U	4	<0.50	10.3
U	5	<0.50	10.2
U	6	n/s	8.9
U	7	n/s	7.0
U	8	n/s	6.5
U	9	n/s	9.1
Note: n/s = not sampled.			

In summary, on the basis of the criterion of 5 mg/L for SPLP lead, the long-term stability of the treated soil appears to have been reduced at the inactive pottery factory by the application of Soil Rescue. The results for treated soil from the trailer park are consistent with long-term stability.

Phosphates

Soil Rescue contains phosphoryl esters used to form metal complexes. Phosphates may be released from the soil into local streams through stormwater runoff. Therefore, two analytical procedures were used to evaluate whether the phosphates in Soil Rescue could be released into the environment. The methods are comparison of the total phosphate concentrations in untreated and treated soils at both sites by SW 846 Method 9056 and comparisons of the concentrations of phosphate that leach from untreated and treated soil when the SPLP test (SW-846 Method 1312) is applied (analyzing the SPLP extract for total phosphates by SW-846 Method 9056).

Table 2-33 lists the total concentrations of phosphate for soil from the trailer park, and Table 2-34 lists the total concentrations of phosphates for soil from the inactive pottery factory. The data from both sites clearly show significant increases in the concentrations of phosphates after the application of Soil Rescue.

Table 2-35 lists the concentrations of SPLP phosphates for untreated and treated soils from the trailer park, and

Table 2-32. SPLP Lead Summary Statistics for Soil from the Inactive Pottery Factory	
Statistic	Data
Mean (mg/L)	8.78
Standard Deviation	1.49
95% UCL	9.76

Table 2-36 lists the concentrations of SPLP phosphates for untreated and treated soil from the inactive pottery factory. The data from both sites also clearly show a significant increase in the concentrations of SPLP phosphates after the application of Soil Rescue.

Table 2-37 displays the estimated means and 95 percent confidence intervals for both sets of data on treated soil from both sites. The estimated mean concentrations of total phosphates were 701.4 mg/kg for the trailer park and 2,145 mg/kg for the inactive pottery factory. The estimated mean concentrations of SPLP phosphates were 49.3 mg/L and 107.7 mg/L for the trailer park and the inactive pottery factory, respectively. On the basis of the data obtained by conduct of analytical procedures, it appears that phosphates from the application of Soil Rescue could leach from the soil, a circumstance that could affect nearby surface water.

The results of the conduct of most of the procedures indicate that Soil Rescue appears to increase long-term stability. However, the results of some of the procedures suggest that Soil Rescue does not increase long-term stability. Long-term stability of soil was indicated for soils treated by Soil Rescue at both test locations, as shown by the analytical results of the MEP, pH, and CEC test procedures. In addition, long-term stability of the soil was indicated at one site, but not at the other, by analytical results of the following tests: lead speciation by sequential extraction, Eh, acid neutralization capacity, and SPLP lead. The analytical results or testing by the lead speciation by SEM (conducted only on soils from the trailer park) were mixed in that some soluble species of lead were reduced, while the organic matter phase of lead was increased. Lead bound to organics can be released if the organic phase is biologically degraded by microbes in the soil. For both locations, long-term stability of soil was not indicated for soils treated by Soil Rescue by the results of separate analyses for total lead by nitric and hydrofluoric acids (higher concentrations of total lead using the hydrofluoric acid method would have indicated long-term

Table 2-33. Total Phosphates Analytical Results for Soil from the Trailer Park			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
C	Composite	<13.2	235
G	Composite	<12.7	1,250
K	Composite	<12.4	580
L	Composite	<12.1	674
M	Composite	<11.5	663
N	Composite	<12.1	1,600
O	Composite	<12.2	680
Q	Composite	<11.5	781
R	Composite	<11.2	192

Table 2-34. Total Phosphates Analytical Results for Soil from the Inactive Pottery Factory			
Experimental Unit	Sampling Location	Untreated (mg/kg)	Treated (mg/kg)
U	1	<12.7	2,180
U	2	<13.4	2,270
U	3	<13.0	1,950
U	4	<13.7	1,620
U	5	<13.5	3,530
U	6	n/s	1,730
U	7	n/s	2,340
U	8	n/s	1,550
U	9	n/s	2,110
Note: n/s = Not sampled			

Table 2-35. SPLP Phosphates Analytical Results for Soil from the Trailer Park			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
C	Composite	<1.0	30.2
G	Composite	<1.0	75.5
K	Composite	<1.0	53.2
L	Composite	<1.0	41.3
M	Composite	<1.0	40.2
N	Composite	<1.0	93.7
O	Composite	<1.0	44.4
Q	Composite	<1.0	52.8
R	Composite	<1.0	27.2
T	Composite	<1.0	34.2

Table 2-36. SPLP Phosphates Analytical Results for Soil from the Inactive Pottery Factory			
Experimental Unit	Sampling Location	Untreated (mg/L)	Treated (mg/L)
U	1	<1.0	96.0
U	2	<1.0	101
U	3	<1.0	89.2
U	4	<1.0	62.0
U	5	<1.0	126
U	6	n/s	66.4
U	7	n/s	107
U	8	n/s	72.6
U	9	n/s	249
Note: n/s = Not sampled			

Table 2-37. Phosphate Summary Statistics			
Location	Data	Mean	95% Confidence Interval
Trailer Park	Total phosphates (mg/kg)	701.4	(430—973)
	SPLP phosphates (mg/L)	49.3	(36—62)
Pottery Factory	Total phosphates (mg/kg)	2,145	(1,757—2,532)
	SPLP phosphates (mg/L)	107.7	(71—145)

stability), total phosphates (significant increases in total phosphates create a higher potential for environmentally damaging releases of phosphates to surface waters), and leachable phosphates as indicated by the SPLP.

2.4.4 Evaluation of S2

Demonstrate that the application of Soil Rescue does not increase the public health risk of exposure to lead.

During the demonstration, it was necessary to remove vegetation with a sod cutter and to prepare the soil for the collection of samples before and after treatment. The activities generated dust that was monitored with real-time devices. Air sampling devices were used to determine the total concentrations of lead in the dust. Accomplishment of S2 was evaluated by collecting air samples through filters during tilling operations and calculating the exposure to lead on the basis of total lead content of the air sampling filters and the length of exposure. The concentration of lead was determined by the nitric acid digestion method described in Section 2.3.1. The exposure calculated was compared with NAAQS for lead, which currently is 1.5 μm^3 of air, averaged over a period of three consecutive months. Table 2-38 lists the exposures calculated for the worker during the demonstration.

The only sample result in the detectable range, 24 mg/m^3 , occurred on September 25, 1998, on the east area sample. The tilling activity at this plot and the corresponding sampling period were 5 minutes in duration. These values extrapolate to a concentration of $9.3 \times 10^{-4} \text{ mg}/\text{m}^3$ over a 3-month period, which is lower than the NAAQS standard. Assuming that the concentration was to remain constant during extended remediation activities; however, the

NAAQS standard would be exceeded after approximately 135 hours. The application of Soil Rescue does not appear to create a significant quantity of dust; however, air monitoring was not conducted during that activity. If it is determined that it is necessary to remove the soil or use other techniques that may generate dust, air monitoring with real-time devices correlated to actual concentrations of lead in the air (for example, high-volume air samplers) and, if appropriate, dust suppression measures should be employed.

2.4.5 Evaluation of Objective S3

Document baseline geophysical and chemical conditions of the soil before the addition of Soil Rescue.

Soil samples collected from the locations at the trailer park and the inactive pottery factory at which the demonstration was conducted were analyzed to determine the soil classification and to determine whether VOCs, SVOCs, or oil and grease were present in the soils.

One soil sample from each of the demonstration sites was analyzed by ASTM Method D 2487-93, Standard Classification of Soils for Engineering Purposes, to determine the soil classification. The soil type for both sites has been identified as sandy silt, an organic clay having low plastic limits and liquid limits of less than 50 percent.

The results of analysis for VOCs did not indicate the presence of any VOCs in the soils at either site. The analysis for SVOCs indicated the presence of the following SVOCs in the soils at the inactive pottery factory: benzo(a)anthracene (0.82 mg/kg), benzo(b)fluoranthene (0.91 mg/kg), benzo(k)fluoranthene (0.77 mg/kg), benzo(a)pyrene (0.69 mg/kg), chrysene (1.0 mg/kg), fluoranthene (1.9 mg/kg), and pyrene (1.9 mg/kg). Those SVOCs typically are found in crude oil, fuel oil, or used motor oil. The soil in that area did show signs of staining that may have been the result of the disposal of a small quantity of waste oil. On the basis of the concentrations detected and the current state regulations governing petroleum releases, it does not appear that the SVOCs present at the site require remediation. The technology developer indicated that the SVOC would not interfere with Soil Rescue. The analytical results for the soil at the inactive pottery factory indicated that oil and grease were present at a concentration of 3,680 mg/kg . The analytical results for the soil at the trailer park did not indicate that oil and grease were present.

Table 2-38. Air Monitoring Results					
Area	Date	Time Sampled (minutes)	Flow Rate (L/minute)	Air Volume (L)	Lead Concentration
Area Sample Southwest	9/22/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample East	9/22/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample Northeast	9/22/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample North	9/22/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample Southwest	9/25/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample East	9/25/98	5	10	50.0	24 $\mu\text{g}/\text{m}^3$
Area Sample Northeast	9/25/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Area Sample North	9/25/98	5	10	50.0	<4.0 $\mu\text{g}/\text{m}^3$
Notes: $\mu\text{g}/\text{m}^3$ = Micrograms per cubic meter of air					

The soil humus fractions (humic acid and fulvic acid) were determined from untreated samples collected from both sites. Humus in soils contributes ligands that can bind with the lead. These concentrations can be used to evaluate whether the humus is contributing to the concentration of the lead species bound to organic fractions. That information is important when a technology uses humic acids to bind the lead. However, since Soil Rescue does not use humic acids to bind the lead, the concentration of humic acids is provided only as a description of the organic matter in the soil. The concentration of humic acid in the soil at the trailer park was 2,400 mg/L, and the concentration of humic acid in the soil at the inactive pottery factory was 1,400 mg/L. The concentration of fulvic acid in the soil at the trailer park was 600 mg/L, and the concentration of fulvic acid at the inactive pottery factory was less than 500 mg/L.

2.4.6 Evaluation of Objective S4

Document the operating and design parameters of Soil Rescue.

On the basis of information obtained through the SITE evaluation from Star Organics and from other sources, an economic analysis examined 12 cost categories for a scenario in which Soil Rescue was applied at full scale to treat soil contaminated with lead at a Superfund site. For the cost estimate, it was assumed that the site was one acre in size and that the treatment was applied to a depth of 6 inches, or approximately 807 cubic yards of soil. The estimate assumed that the soil characteristics and lead concentrations of lead at the site were the same as those encountered during the CRPAC evaluation. With those assumptions, the total costs were estimated to be \$32,500

per acre or \$40.27 per yd^3 . Costs for application of Soil Rescue may vary significantly from that estimate, depending on site-specific factors.

2.5 QUALITY CONTROL RESULTS

The overall quality assurance (QA) objective for the SITE program demonstration, as set forth in the QAPP, was to produce well-documented data of known quality as measured by the precision, accuracy, completeness, representativeness, and comparability of the data, and the conformance of the data to the project-required detection limits (PRDL) for the analytical methods. Specific QA objectives were established as benchmarks by which each of the criteria was to be evaluated. Section 3.0 of the QAPP presented the QA objectives for the critical parameters.

This section discusses the quality control (QC) data with respect to the QA objective of the project for critical parameters. The results, and those for noncritical parameters, can be found in the unpublished TER for this SITE demonstration (Tetra Tech 2001). The TER is available upon request from the EPA work assignment manager (see Section 1.4 for contact information).

QA objectives for laboratory analysis of the critical parameter bioavailable lead were evaluated on the basis of analytical results from matrix spike samples and matrix spike duplicate samples (MS/MSD), blank spikes, laboratory control samples (LCS), reagent blanks, bottle blanks, and calibration criteria. QA objectives for laboratory analysis of the critical parameter TCLP lead were evaluated on the basis of MS/MSDs, LCS/LCSD, and method

blanks. Table 7-1 of the QAPP summarizes the internal acceptance criteria for laboratory QC samples, as well as corrective action procedures for the demonstration.

2.5.1 Completeness

The QA objective for data completeness specified by the QAPP is that 100 percent of all planned measurements will be obtained and will be valid. As discussed in Section 3.1, SITE program personnel did not collect an equipment and field blank during the sampling of treated soil for analysis for bioavailable lead. Analytical results for the equipment and field blanks for untreated soils and subsequent long-term monitoring blanks did not indicate cross-contamination as a result of sample collection or shipping procedures. Therefore, the deviation should not affect overall data quality. All the soil samples specified in the QAPP for TCLP lead analysis were collected and analyzed. All samples were analyzed within the holding times specified in the QAPP, and all the TCLP lead data were considered usable. Therefore, the critical parameters of bioavailable and TCLP lead data are considered 100 percent complete.

2.5.2 Comparability and Project-Required Detection Limits

On the basis of consistent implementation of a reference method, data on critical parameters (bioavailable lead and TCLP lead) for samples of untreated and treated soil are considered to be comparable. As specified by the QAPP, the University of Colorado used the SBRC's, SIVM to analyze soil samples for bioavailable lead, and Quanterra used SW-846 Method 1311 (EPA 1996) to analyze soil samples for concentrations of TCLP lead. The PRDLs specified in Table 3-1 of the QAPP were achieved for all samples collected during the demonstration.

2.5.3 Accuracy and Precision

Accomplishment of QA objectives for accuracy and precision were evaluated on the basis of MS/MSD percent recoveries and relative percent differences (RPD). Percent recovery and RPD values for LCS/LCSD and blank spike (BS) samples also supported QA objectives for accuracy and precision.

All the assessments of precision and accuracy for the bioavailable lead data, including the RPD of the duplicates and the percent recoveries of the MS and BS analyses, were within the limits specified in the QAPP. Concentration levels for spiking met the criteria specified in the QAPP for

all analyses. Appendix B presents the QC data for the critical and noncritical parameters.

One TCLP lead MS/MSD sample had a percent recovery of 124 percent, which is outside the acceptable range of 80 to 120 percent. The batch of samples for which the MS/MSD analysis was performed were all samples of untreated soil. Therefore, the deviation should have no effect on the overall quality of the data for the demonstration. The data on untreated soil are not used to determine whether the technology can meet objective P1, which is to reduce the TCLP lead concentration to a level lower than the alternative UTS lead in soil of 7.5 mg/L. The percent recovery of the LCS/LCSDs were all within the acceptable range of 80 to 120 percent. All the RPDs for the MS/MSD and LCS/LCSD samples were less than 20 percent and therefore were acceptable.

2.5.4 Representativeness

The University of Colorado analyzed method blank samples for bioavailable lead to confirm the representativeness of the data on bioavailable lead by determining whether any lead might have been introduced during preparation and analysis of the samples. The levels of lead in the method blank samples did not exceed the criteria set forth in the QAPP for method blanks, which is 25 µg/L. Therefore, the method blank analyses do not indicate that laboratory contamination introduced detectable concentrations of the critical parameter bioavailable lead into any of the samples, and the reported concentrations of the critical parameter bioavailable lead appear to be representative of actual concentrations in the soil samples, as indicated by the available QC data.

Quanterra analyzed method blank samples for TCLP lead to confirm the representativeness of the TCLP lead data by determining whether any lead might have been introduced during sample preparation and analysis of the samples. Quanterra did not detect any TCLP lead in any of the method blanks at levels higher than the PRDL of 0.50 mg/L. Therefore, the method blank analyses do not indicate that laboratory contamination introduced detectable concentrations of the critical parameter TCLP lead into any of the samples, and the reported concentrations of the parameter TCLP lead appear to be representative of actual concentrations in the soil samples, as indicated by the available QC data.

Tetra Tech prepared equipment blank samples and field blank samples to determine whether any lead might have been introduced by sample collection, handling, and

packaging procedures. Section 2.5.1 of the TER summarizes the blank sample preparation techniques. No lead was detected in any of the blank samples at levels higher than the PRDL of 100 µg/L.

The University of Colorado analyzed the equipment blank and field blank samples for bioavailable lead to confirm the representativeness of the data on bioavailable lead by determining whether any bioavailable lead might have been introduced during sample collection, handling, and packaging. The University of Colorado did not detect any bioavailable lead in any of the equipment and field blanks at levels higher than the PRDL of 100 µg/L. Therefore, the results of analysis of the equipment and field blanks do not indicate that sample collection, handling and packaging

procedures introduced detectable concentrations of the critical parameter bioavailable lead into any of the samples.

Quanterra analyzed the equipment blank and field blank samples for TCLP lead to confirm the representativeness of the TCLP lead data by determining whether any lead might have been introduced during sample collection, handling and packaging. Quanterra did not detect any TCLP lead in any of the equipment and field blanks at levels higher than the PRDL of 0.50 mg/L. Therefore, the analysis of equipment and field blanks do not indicate that sample collection, handling and packaging procedures introduced detectable concentrations of the critical parameter TCLP lead into any of the samples.